STUDIES OF ION-EXCLUSION ON GLYCEROL-SODIUM CHLORIDE-WATER SYSTEM USING RESIN DOWEX 50 W

A Thesis Submitted
in Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

46887

By
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to the

DEPARTMENT OF CHEMICAL ENGINEERING
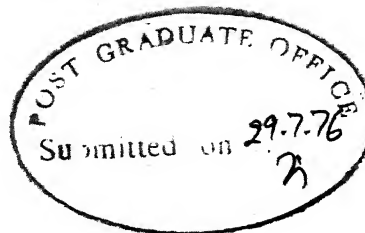INDIAN INSTITUTE OF TECHNOLOGY KANPUR
JULY, 1976

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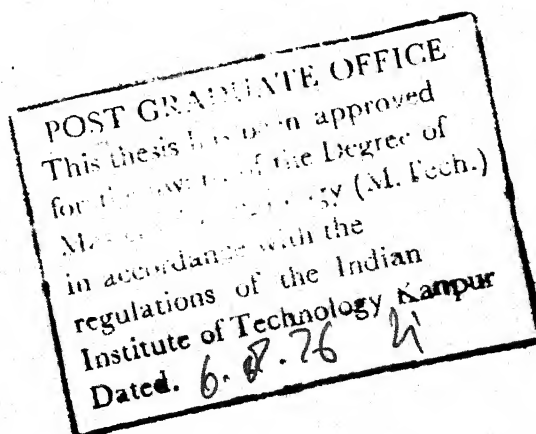
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Dated. 6. 8. 76

ACKNOWLEDGEMENTS

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NOMENCLATURE

a_s Activity in the solution phase

a_R Activity in the resin phase

C_e Effluent concentration, gram/cc for NaCl, weight per cent for glycerol

C_f Feed concentration, gram/cc for NaCl, weight per cent for glycerol

C_{s_i} Concentration of solute in internal liquid phase of the resin, gram/cc

C_{s_o} Concentration of solute in external liquid phase of the resin, gram/cc

D.V.B.   Divinyl Benzene

E   Donnan Potentials

F   Faraday constant

h   Height of resin bed, inches

H.E.T.P.   Height equivalent to theoretical plate, inches

K   Activity coefficient ratio

K_d Distribution coefficient ratio

m   Molality

n   Valence of Ion

n_d^{20} Refractive index at $20^{\circ}C$

P   Number of theoretical plates

R   Universal gas constant

T   Temperature, $^{\circ}K$

V_{liquid} Interstitial volume of the resin, cc

V_{liquid}^{r} Occluded volume of the resin, cc

V_R^r Volume of resin net work, cc

V_T Total bed volume, cc

V_e Effluent volume, cc

V_F Feed volume, cc

W   Half width of the elution curve at ordinate value of 1/e of the peak concentration (where e is the log base). W is measured in the same volume unit as V_{liquid}, V_{liquid}^r and V_m

W'   Same as W except that W' is measured in column volume (liquid volume held between particles in column)

C'   Distribution ratio of a solute in any plate

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SYNOPSIS

Ion-exclusion is a chromatographic technique for separating materials of different ionic-activity and molecular size by means of ion-exchange resins. Ionic materials are largely excluded from the resin because of the high ionic concentration within the resin beads. Ion exclusion can be used to separate two or more solutes as long as the solutes in question have different distribution coefficient ratios.

In the present study the separation of ionic impurities (mainly sodium chloride) from crude glycerol was carried out by a strongly acidic cation exchange resin viz. Dowex 50W-X8, Na^+ form, 50-100 mesh. The effect of variable viz. temperature, flow rate, feed volume, cross-linkage, resin particle size and feed concentration were investigated. A recycle method has also been studied for the improving the separation and concentration of the product.

It has been found that higher temperature, lower flow rate, small particle size, low cross-linkage, small feed volume and low ionic concentration give good separation. By using the recycle technique, it is possible to obtain an almost pure white sample of glycerol in concentration equal to or greater than the feed concentration.

The efficiency of an ion-exclusion column was determined by the elution of a single component (glycerol) from Dowex 50W

and analysis of the elution curve was used to determine
the H.E.T.P. The dependence of H.E.T.P. on various factors
such as temperature, flow rate, feed volume, cross-linkage
and particle size was established.

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CHAPTER 1

INTRODUCTION

Ion-exclusion process is a unit operation which utilizes ion-exchange resins to separate solutes of different ionic activity without the use of chemical regenerants. The separation is dependent upon the physical and chemical properties of the resin as well as the solutes, and no net ion-exchange takes place.

It has been established that when an ion-exchange resin in a particular ionic form is placed in a dilute aqueous solution of an electrolyte containing the same ion, the resulting concentration of the electrolyte is lower within (the aqueous portion of) the resin phase in comparison to the concentration in the surrounding solution. This may be explained by the Donnan-membrane equilibrium set up in such a manner that the electrolytes are primarily excluded from the resin phase. Ion-exclusion, therefore, would be more effective to separate ionic from non-ionic solutes in polar solvents. However, ion-exclusion can be used to separate two or more solutes as long as the solutes in question have different resin-liquid distribution coefficients. The distribution coefficient (K_d^i) of a solute may be defined as the ratio of the concentration of solute in the internal liquid phase of the resin to the concentration of the solute in the liquid

phase external to the resin. The difference in distribution coefficient for different solutes is the basis of ion-exclusion separations and the degree of separation represents the magnitude of this difference.

The distribution coefficient would also determine the sequential order for the solutes to appear in the effluent stream. Thus, when an aqueous solution of two or more solutes is percolated through an ion-exclusion column, a separation or partial separation of the solutes occur and they appear as separate fractions in the effluents. The solute having the lowest value of distribution coefficient will elute first and so on. The distribution coefficients of ionic-solutes usually have relatively small values (0.1 to 2.0) while for non-ionic solutes they range from 0.4 to 2.0. In the case of an aqueous solution of ionic and non-ionic solutes, the ionic solutes by virtue of their lower distribution coefficients would appear in the effluent prior to the non-ionic solutes.

In ion-exclusion no net exchange of ion takes place with the result that regeneration of the resin is not required and it is in this respect that ion-exclusion differs from ion-exchange. The ion-exclusion has many advantages over the conventional ion-exchange such as:

1. Ion-exclusion is most suitable at higher ionic concentration, where ion-exchange is prohibitive in cost.

2. Ion-exclusion can be applied for separation of a wide range of solute materials which cannot be separated economically by conventional ion-exchange.

3. The regeneration cost is saved.

4. The only operating cost is for pumping and water requirement.

5. It is also a simple process to operate because there is no regeneration step.

6. It can separate two or more ionic or non-ionic solutes.

7. It is also advantageous for deionizing pH-sensitive materials where the use of H^+ or OH^- form of resin is detrimental.

8. The simplicity of ion-exclusion equipment and its control is reflected in its low cost operation.

Potentialities of this relatively new process of ion-exclusion have been utilized for the separation of solutes mixtures of ionic-non ionic, ionic-ionic and non ionic-non ionic character and the process appears to be promising. Some of the systems with water as solvent, investigated for suitability of ion-exclusion process, using Dowex 50W (a polystyrene sulphonic acid type resin marketed by Dow Chemical Co., U.S.A.) are given below.

1. Sodium chloride-ethylene glycol

2. Sodium chloride - glycerol

3. Hydrochloric acid - acetic acid

4. Sodium chloride - ethanol

5. Sodium chloride - formaldehyde

6. Hydrochloric acid - acetic acid

7. Acetone-formaldehyde

8. d-glucose-methanol

9. Sodium chloride - amines

10. Sucrose-sodium chloride

11. Acetic acid - acetone

12. Sucrose recovery from beet molasses

Besides the above, one multicomponent system (sucrose-glycerol-triethylene glycol-phenol) has also been investigated.

Glycerol is a byproduct of soap industry and contains mainly sodium chloride besides other impurities including water. The proximate analysis of the crude product is thus:

Glycerol   =   82-86 per cent weight

Sodium chloride = 7-10 per cent weight

Dissolved soap
Coloring matter
Water
Sodium carbonate   = 5-10 weight per cent

The glycerol-sodium chloride-water system can be used for initial study as a model approximating crude glycerol.

Glycerol is extensively used as an ingredient in products where highest degree of purity is essential. Crude glycerol is refined by evaporation followed by distillation. Another practice is to purify crude glycerol by deionization followed by evaporation. Ion-exclusion can also be used to recover pure glycerol from the soap lyes. However, the economic feasibility of ion-exclusion can be established only after laboratory investigations. The present work, therefore, has been directed towards studies on the purification of glycerol from sodium chloride-glycerol-water system.

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CHAPTER II

LITERATURE REVIEW

II-1. Review of Previous Work:

Bauman and Eichhorn (1) have shown that when an
ion-exchange resin is placed in a dilute solution of an
electrolyte, the concentration of that electrolyte is lower
within the aqueous portion of the resin phase than in the
surrounding solution. They have shown it to be true with
Dowex 50W-X12, H^+ form after equilibration with standard
HCl.

Gregor et al. (2) have made similar studies on
Dowex 50 with urea and ethyl acetate as solute. They showed
that the relative inside and outside concentration of non-
ionic material is dependent on the ionic form of the resin,
degree of cross-linkage, mesh size and the equilibrium
temperature. Time is a factor to be considered in reaching
equilibrium.

Wheaton and Bauman (3) employed ion-exclusion
technique for $HCl-CH_3COOH$ system using Dowex 50W-X8, 50-100
mesh, H^+ form. The resin bed dimension were approximately
(1.5 cm x 62 cm) with a fritted glass filter support. Samples
were collected by use of a Technicon fraction collector,
which collected a predetermined number of drops in successive
test tubes. They also studied the effect of operating

variable viz. temperature, cross-linkage, feed volume, particle size and feed concentration on the separation of these solutes.

Bauman and Wheaton (4) have studied the following systems using the Dowex 50-X8, 50-100 mesh. Resin bed dimensions were approximately (1.5 cm x 55 cm).

a. d-glucose-methanol   Dowex 50-X8, 50-100 mesh, H^+ form

b. Sodium chloride-Alcohol   Dowex 50-X8, 50-100 mesh, Na^+ form

c. Sodium chloride-xylose   Dowex 50-X8, 50-100 mesh, Na^+ form

d. Sodium chloride-glycols   Dowex 50-X8, 50-100 mesh, Na^+ form

e. Sodium chloride-amines   Dowex 50-X8, 50-100 mesh, Na^+ form

f. Formaldehyde-acetone   Dowex   1-X8, 50-100 mesh, SO_4^{-2} form

g. d-glucose-acetone   Dowex 50-X8, 50-100 mesh, Na^+ form

h. Acetic acid-acetone   Dowex 50-X8, 50-100 mesh, H^+ form

They also undertook an investigation of the multi-component system i.e. sucrose-glycerine-triethylene glycol-phenol using Dowex 50-X8, 50-100 mesh, Na^+ form. The feed consisted of 5 cc of solution containing 5 per cent each of sucrose (K_d = 0.24) glycerin (K_d=0.49), triethylene glycol (K_d=0.74) and phenol (K_d=3.08).

Simpson and Wheaton (5) studied the efficiency of an ion-exclusion column for the elution of ethylene glycol in an aqueous mixture with sodium chloride as the second component. The experimental data were obtained with a resin bed volume of approximately 100 ml (column 0.6" x 22"). Flow rates

were controlled by a stop cock at the bottom of resin bed while maintaining a constant head. Simpson and Wheaton (6) later developed a Recycle method for ion-exclusion technique for the same system. The laboratory column (2.75"x15") was filled with approximately 1460 ml of Na^+ form of Dowex 50-X12, 50-100 mesh. They also studied the effect of sodium chloride concentration on the peak and average concentration of the ethylene glycol using their recycle method.

Asher and Simpson (7) studied the purification of glycerol on a laboratory scale by ion-exclusion process. They showed that by a method of recycle, it was possible to obtain a glycerol product of near feed concentration while reducing the ionic content to a low value. It was found that separation improved by the use of elavated temperatures. The optimum feed volume for this method of operation was 30-35 per cent of the resin bed volume. For maximum product concentration the feed was shown to contain 30-35 per cent glycerol. The salt concentration did not significantly affect the separating capacity of the resin although a higher salt concentration tended to increase the product concentration.

Asher (8) has reported sugar recovery from molasses by ion exclusion using Dowex 50W-X4, 50-100 mesh. He was also successful in decolorizing a 30 per cent solution of molasses. The bulk of the colour was eluted with the ionic components. Similar results were found using a dark solution of a raw cane sugar.

Prillipp and Keller (9) found that the interstitial volume for a uniformly spherical resin sample was approximately 38 per cent and the occluded volume approximately 37 per cent for Dowex 50W-X8, 50-100 mesh, Na^+ form. The values of distribution coefficient for sodium chloride and glycerol are 0.19 and 0.59 respectively.

The difference in K_d value was utilized for purification of crude glycerol and the effect of variables such as temperature, flow rate, feed volume and feed concentration were investigated.

Shurts (10) has measured the ion-exclusion equilibria for the system glycerol-sodium chloride - water - Dowex 50-X8. He showed that the distribution coefficient of glycerol and sodium chloride varies with glycerol concentration, sodium chloride concentration and total concentration.

Walter (11) has measured the ion-exclusion equilibria for the system sucrose-KCl-H_2O-Dowex 50W-X4. In this study the effect of temperature and salt concentration on equilibrium in the system at sucrose concentration from 0 to 60 per cent weight and KCl concentration of 0,1,3 and 6 weight per cent have been investigated.

Stark, Lowe and Schultz (12) have attempted sucrose recovery from beet molasses using Dowex 50W-X4, 50-100 mesh, K^+ form on a bench scale. They also carried out engineering analysis and provided cost projection of the process.

Sutherland and Mounffest (13) reported data on measurement of ion-exclusion equilibria for the system sucrose-NaCl-H_2O-Dowex 50W-X4. The phase equilibria were investigated at $25^{\circ}C$ for the range of weight concentration 0-3 per cent sodium chloride and 0-60 per cent sucrose. The material balance technique was used.

Zievers (14) carried out partial purification of a low purity syrup using ion-exclusion technique in a column of Dowex 50W-X4, 50-100 mesh. A reasonably pure product with relatively high colour was obtained. Some of the colorants in the ion-exclusion product were not easily removed by the an ion exchange resin (IRA 401S).

A review of all these investigations leads to the following general conclusion about some aspects of the ion exclusion process.

II-2. <u>Choice of Resin</u>:

Almost any type of the ion-exchange resin may be used for the ion-exclusion process. However the more strongly ionized ion-exchange resins are preferred for the most efficient and economical operations. It is essential that the resin be in the same ionic form as the ionic material which is being separated.

For the purification of glycerol the principal ionic impurity is sodium chloride, so the resin chosen should be either in Na^+ form of a cation exchanger or Cl^- form of an

anion exchanger. The successful application of the ion-exclusion technique requires an ion-exchange resin which will most satisfactorily meet the following requirements.

1. It should be highly ionized, chemically, mechanically and thermally stable, cheaper in price with high capacity, besides being non-reactive with any of the components in the feed mixture.

2. It gives low colour throw during cycles of operations.

3. The fixed ionic concentration inside the resin particle must be high and so a resin with high degree of cross-linkage is desirable.

4. The water content of the resin particles must be high enough to given an appreciable capacity for holding the non-ionic component.

5. The porosity of resin spheres must be such that equilibrium between resin particles and the solute should be rapidly attained i.e. diffusion of the solute should be quick.

Dowex is a trade name of the resins manufactured by the Dow Chemical Co., Midland, Michigan, U.S.A. The following resins of the company satisfy the requirements. However they should be in the appropriate chemical form.

1. Dowex 50W

2. Dowex 1

Dowex 50W:

Is a generic name for a series of strongly acidic styrene base cation exchange resin containing sulphonic acid group. The cross-linkage is indicated by symbol X followed by a number - say X-4 which denotes a resin with 4 per cent divinyl benzene content.

Dowex 1:

Is a generic name for a series of strongly basic anion exchange resin containing quarternary amino group.

The performance of Dowex 50W-X4 and Dowex 1-X4 for ion exclusion were reported by Asher and Simpson (7) to be comparable. However, the thermal, mechanical and chemical stability of the strongly basic anion exchange resin have been shown to be poor. They are also much more expensive. Dowex 50 was thus chosen for the purposes of this study.

Cross Linkage:

Simpson and Wheaton (5) pointed out that 4 to 12 per cent cross-linkage resins give the best separations for most systems. Resin with lower cross linkage (< 4 per cent) swell and shrink to a greater degree with changing concentration, especially with ionic materials, thus causing a changing bed volume and non uniform pressure drop across the bed. This makes the operation more difficult. Other important practical considerations may also be mentioned:

1. The lower cross-linkage resins have fewer exchange sites per unit volume and hence a smaller exclusion factor.
2. The resin particles are mechanically less stable, have a higher attrition loss and gives a more dilute product.
3. They swell considerably and are soft and gelatinous.
4. Favour more rapid diffusion of the non-ionic component but poorer exclusion of the ionic component.

Cross-linkage higher than 12 per cent has corresponding advantages but is not suitable because the porosity is low and hence the diffusion rates are poor and disadvantageous. If used with a resin of higher cross linkage not only ionic but non-ionic components of the bigger molecular dimensions also would be excluded from the resin by size effect alone. Therefore, the range of cross-linkage used was from 4 to 12 per cent.

Particle Size:

The particle size of an ion-exchange bead may be controlled by controlling the condition during suspension polymerization. The effect of particle size on the ion-exclusion separation is also very pronounced. One of the requirements for the ion-exclusion separation is that equilibrium must be attained rapidly. The finer-mesh resin favour rapid attainment of the equilibrium resulting in improved separation. However, the finer mesh resins have the disadvantage of increasing pressure drop through the bed and slower flow rate.

II-3. Equilibrium:

The time required to reach equilibrium is dependent on cross-linkage and the particle size of the resin and on the nature of the solutes.

A comparison of time required to reach equilibrium using the Dowex 50W-X8, at 20-50 mesh, 50-100 mesh and 200-400 mesh with 5 per cent ethylene glycol has been reported by Bauman and Wheaton (4) and shown in Figure 1. The time required to reach the equilibrium for fine resin is less as compared to resin with coarse particle size.

Similarly for a given resin size, equilibrium is more rapidly attained with the lower-cross linkage resin as shown in Figure 2. The same relationships should hold true by and large for any solute. The order of magnitude, however, may differ appreciably.

II-4. Distribution Coefficient:

The distribution coefficient of a solute is defined as the ratio of the concentration of the solute within the sorbent (resin beads) and in the solution surrounding the resin beads.

$$K_d = \frac{\text{Concentration of solute in internal liquid phase of the resin}}{\text{Concentration of solute in external liquid phase surrounding the resin}}$$

$$K_d = \frac{C_{s_i}}{C_{s_o}}$$

where C_{s_i} and C_{s_o} are defined on a weight/volume basis i.e. gram/ml. Thus
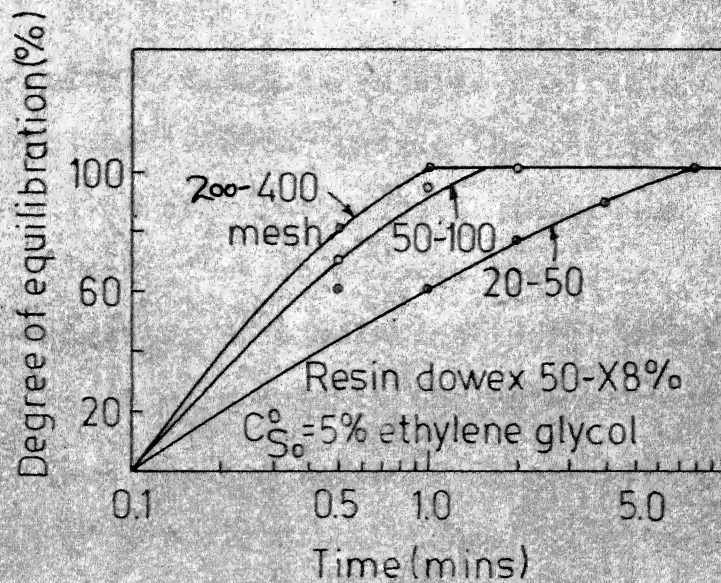
Fig. 1 - Equilibration rate vs. particle size.
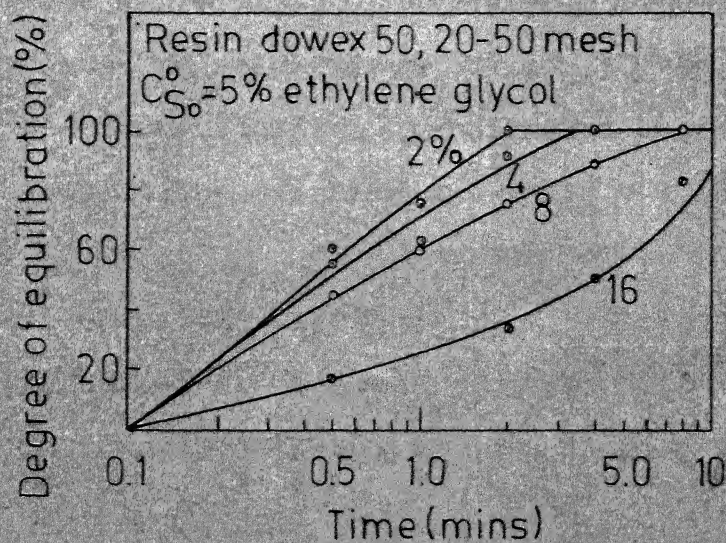
Fig. 2 - Equilibration rate vs. cross linkage.

$$C_{s_i} = \frac{M_{s_i}}{V_{s_i} + V_{w_i}} \quad \text{and} \quad C_{s_o} = \frac{M_{s_o}}{V_{s_o} + V_{w_o}}$$

V_{s_i} = volume of solute in side the resin

V_{w_i} = volume of water inside the resin

V_{s_o} = Volume of solute outside the resin

V_{w_o} = Volume of water outside the resin

M_{s_i} = Weight of solute in the inside portion

M_{s_o} = Weight of solute in the outside portion

Bauman and Wheaton (4) have described a method for the determination of the distribution coefficient (given in Appendix (B)).

Gregor et al. (2) have shown that the relative distribution of solute within the resin and outside the resin (as reflected by the distribution coefficient) is dependent on a number of factors including types and ionic form of the resin. Shurts (10) has found that the distribution coefficients vary with the concentration of ionic, non-ionic and total concentration.

Table 1 gives the distribution coefficient (K_d) values for a number of solutes. These values high light the variation resulting from changes of resin types and ionic form of the resin.

Shurts (10) showed that for Dowex 50W $-$ Na$^+$ form the distribution coefficient of sodium chloride and glycerol concen. vary with glycerol concentration, sodium chloride concentration and total concentration. The data are presented in Figures 3 and 4.

TABLE 1

DISTRIBUTION OF COEFFICIENT SOLUTES

$$C_{s_o}^o = 0.05$$

Solute	Resin	K_d
Ethylene glycol	Dowex 50-X8, H^+ form	0.67
Ethylene glycol	Dowex 50-X8, Na^+ form	0.63
Formaldehyde	Dowex 50-X8, H^+ form	0.59
Formaldehyde	Dowex 1-X7.5, Cl^- form	1.06
Formaldehyde	Dowex 1-X8, SO_4^{-2} form	1.02
Glycerol	Dowex 50-X8, H^+ form	0.49
Glycerol	Dowex 1-X7.5, Cl^- form	1.12
Glycerol	Dowex 50-X8, Na^+ form	0.56
Phenol	Dowex 50-X8, H^+ form	3.08
Phenol	Dowex 1-X7.5, Cl^- form	17.70
Acetone	Dowex 50-X8, H^+ form	1.20
Acetone	Dowex 1-X7.5, Cl^- form	1.08
Acetone	Dowex 1-X8, SO_4^{-2} form	0.66
Sucrose	Dowex 50-X8, H^+ form	0.24
d-Glucose	Dowex 50-X8, H^+ form	0.22
Acetic Acid	Dowex 50-X8, H^+ form	0.71
Methanol	Dowex 50-X8, H^+ form	0.61
Xylose	Dowex 50-X8, Na^+ form	0.45
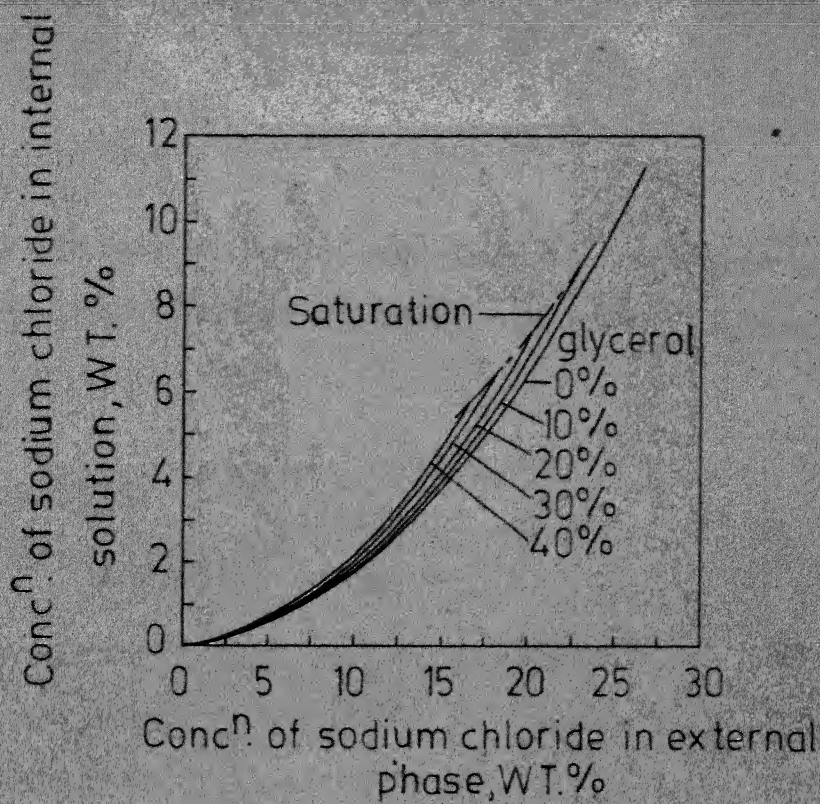
Fig. 3 - Sodium chloride equilibrium.
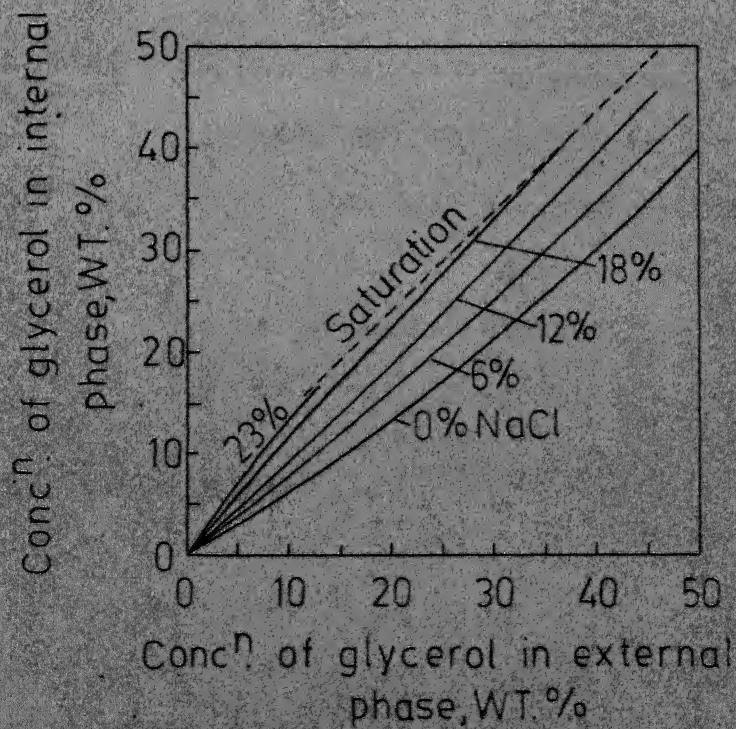
Fig. 4 - Glycerol equilibrium.

On the basis of his experimental data Shurts developed an empirical equation for glycerol and sodium chloride solutes as under:

For glycerol:

$$Y_G = 0.5687\ X_G + 0.004472\ X_G^2 - 0.000113\ X_G^2\ X_S$$
$$+ 0.023023\ X_G X_S + 0.000661\ X_G^{0.4}\ X_S^2$$

Standard residual error = 0.149

For sodium chloride:

$$Y_S = 0.0286\ X_S + 0.01408\ X_S^2 + 0.0001102\ X_G X_S^2$$

Standard residual error = 0.066

where,

X_G and X_S = weight per cent in external phase of glycerol and sodium chloride respectively.

Y_G and Y_S = weight per cent of glycerol and sodium chloride in the aqueous resin phase (i.e. in the aqueous phase within the resin bead and excluding the resinous matrix).

II-5. Column and Bed Height:

The column used in the ion exclusion should be set up in such a way that solutions pass through the column in a piston like manner and any undue disturbance of the band boundaries is avoided. Height to diameter ratio of about 100:1 have been used.

The column height should be chosen such that it allows for 60 per cent bed expansion during back wash operation. It is to be expected that as that bed height increased, the elution curve would be sharper and the separation would improve. On the other hand larger quantity of ion-exchange resin would be needed and that the flow resistance would be higher.

The dead volume (liquid volume below the resin support) should be kept as small as possible, so that mixing of consecutive portions of the effluent is kept at the minimum.

Fraction Collection:

The sample can be collected in test tubes by use of a fraction collector which delivers a predetermined number of drops into each tube before moving on to the next. However, the instrument has one drawback; individual droplets may vary in size with surface tension as the quantity and kind of solute are changed. The more recent practice is to collect the samples in calibrated test tubes and this was followed in the present work.

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CHAPTER III

THEORY AND COLUMN ANALYSIS OF
ION – EXCLUSION

III.1 Theory of Ion-Exclusion:

In separating an electrolyte i.e. sodium chloride from a nonelectrolyte such as glycerol by ion-exclusion, the role of the ion-exchange resin appears to be dual. With regard to the electrolyte the resin may be described as a second solution phase separated from the outside solution by a hypothetical membrane which is largely permeable to all the species present except the exchangeable ions (Na^+) attached to fixed ionic group (SO_3^-) of the resin. To the non-electrolyte the resin behaves as a solid sorbent phase in contact with the solution phase.

Electrolyte Exclusion:

Let us consider an ion exchange bead of sulphonic acid type resin (Dowex 50) in Na^+ form kept in contact with a dilute solution of sodium chloride. The equilibrium set up can be described by Donnan theory. The resin will take on a negative charge with respect to the solution phase. The presence of negative charge is because of a slight excess migration of cation (Na^+) into the solution and of slight excess migration of anion (Cl^-) into the resin. This results is an accumulation of positive charge in the solution and of negative charge in the ion-exchange resin. Both migrations are diffusion controlled

phenomena. The first few ions which diffuse thus build up an electrical potential difference between the two phase. This so called 'Donnan Potential' pulls cations (Na^+) back into the negatively charged ion-exchanger and anions (Cl^-) back into the positively charged solution. Finally an equilibrium is established in which the tendency of ions to level out the existing concentration difference is balanced by the action of the electric feed. In the ion-exchanger, the counter-ion (Cl^-) concentration thus remain much higher and the co-ion (Na^+) concentration much lower than in the external solution.

The magnitude of the potential difference E, between the solution and resin phase due to excess migration of anion into the resin phase is given by the Donnan potential.

$$E = \frac{RT}{n\,F} \ln \frac{a_{Cl^-_S}}{a_{Cl^-_R}} \tag{3.1}$$

where

F = Faraday constant

n = Valence of ion (Cl^-)

$a_{Cl^-_S}$ and $a_{Cl^-_R}$ = Activity of Cl^- in the solution and the resin phase respectively

R = Universal gas constant

T = Temperature in degree Kelvin

At equilibrium the potential will counter balance the diffusion force and thus prevent further concentration-difference controlled migration of electrolyte ions between

the phases. Further, at equilibrium the activity product
of the mobile electrolyte species in the resin phase will be
equal to the activity product in the solution phase. For the
particular case of a 1-1 electrolyte the following relationship
holds good.

$$(\gamma m)_{Na_R^+} \ (\gamma m)_{Cl_R^-} = (\gamma m)_{Na_S^+} \ (\gamma m)_{Cl_S^-} \qquad (3.2)$$

where

γ = single ion activity coefficient

m = ion molality

The suffix S and R represent the solution and resin
phase respectively.

Bearing in mind that amount of excess anion in the
resin and excess cation in the solution are extremely small
and in order for the principle of electroneutrality to be
obeyed.

$$m_{Na_S^+} = m_{Cl_S^-} \qquad (3.3)$$

$$m_{Na_R^+} = m_{Cl_R^-} + m_{R_R^-} \qquad (3.4)$$

where $m_{R_R^-}$ is the molality of the fixed ionic sulphonic acid
group.

From equation 3.2

$$m_{Cl_R^-} = \left(\frac{m_{Cl_S^-} \times m_{Na_S^+}}{m_{Na_R^+}} \right) \left(\frac{\gamma_{Na_S^+} \ \gamma_{Cl_S^-}}{\gamma_{Na_R^+} \ \gamma_{Cl_R^-}} \right)$$

Let us put

$$K = \frac{\gamma_{Na^+_s}\ \gamma_{Cl^-_s}}{\gamma_{Cl^-_R}\ \gamma_{Na^+_R}}$$, which we may call ionic activity

coefficient ratio

$$m_{Cl^-_R} = \left(\frac{m_{Cl^-_s} \times m_{Na^+_s}}{m_{Na^+_R}}\right) K \tag{3.5}$$

Substituting equation 3.3 in equation 3.5

$$m_{Cl^-_R} = K\left(\frac{m_{Cl^-_s}\ m_{Cl^-_s}}{m_{Na^+_R}}\right) = \frac{K\ m^2_{Cl^-_s}}{m_{Na^+_R}}$$

or

$$K\ m^2_{Cl^-_s} = m_{Cl^-_R} \times m_{Na^+_R}$$

We may set $K = 1$ which is a valid approximation for dilute

electrolyte solution. Hence,

$$m^2_{Cl^-_s} = m_{Cl^-_R}\ m_{Na^+_R}$$

Since from equation 3.4

$$m_{Na^+_R} \text{ must be greater than } m_{Cl^-_R} \text{ , so}$$

$$m_{Cl^-_s} > m_{Cl^-_R}$$

In other words the concentration of NaCl is greater
on the solution side (i.e. the phase free of the non diffusible
ion) as compared to the resin phase.

Referring back to equation 3.1, higher the Donnan
potential (in absolute value), stronger is the exclusion i.e.
smaller the electrolyte uptake. The donnan potential, depends
on the ionic concentration and valence. Equilibrium is attained

when the action of the Donnan potential balances the tendency
of counter ions to diffuse out into the solution. The donnan
potential is greater when the concentration difference between
the ion exchanger and the solution is larger. The absolute
value of the Donnan potential increases with decreasing external
and increasing internal counter ion concentration and so does
the efficiency of electrolyte exclusion. The internal counter
ion molal concentration is high when the resin is highly cross
linkage. In summary, the efficiency of electrolyte exclusion
increases with:

  (i) low solution concentration

  (ii) higher capacity of the resin

  (iii) higher D.V.B. per cent

  (iv) choice of counter ion of low valence and co-ions of
       high valence.

Non-Electrolytes (Glycerol)

The non electrolyte in contact with the ion exchange
resin will see no Donnan potential barrier to the solution
within the resin phase because of absence of interactions of any
kind. It is to be expected that the concentration of the solute
in the sorbent and in the external solution are equal. It is
surprising that such a behaviour is really formed. Various
interaction cause deviation from ideality and in practice K_d
is seldom close to unity in most cases.

III-2. Column Analysis

The first column analysis was carried out by Simpson and Wheaton (5). The resin bed consists of three parts

(1) the interstitial volume or the liquid volume between the resin beads (V_{liquid}),

(2) the occluded volume or the volume of liquid within the beads (V_{liquid}^r), and

(3) the volume of the resin network or the solid volume. The total volume is denoted by V_T
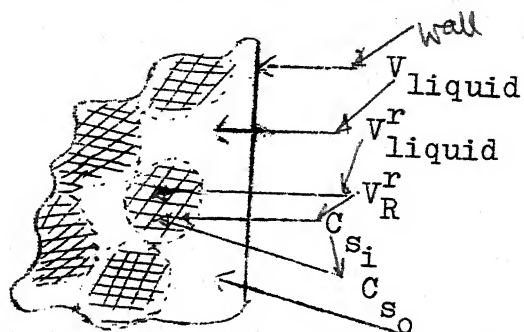
$$V_T = V_{liquid} + V_{liquid}^r + V_R^r$$

$$K_d = \frac{C_{s_i}}{C_{s_o}} \qquad (3.6)$$

Magnified Section of a Column

Because of high ionic concentration within the resin bead and the organic nature of the resin, the character of a solution inside the resin phase is different from the outside, generally causing an unequal distribution of a solute between the two phases.

If a feed solution was fed into a resin bed until a solute first appeared in the effluent, the concentration (C_{s_o} in the interstitial volume (V_{liquid}) would be equal to the initial concentration (C_F) of the solute in the feed. At this point the sum of the $C_{s_o} V_{liquid}$ and $C_{s_i} V_{liquid}^r$ (product of the

concentration and volume inside the resin) is equal to the initial feed concentration multiplied by feed volume (V_F), since the effluent volume is equal to the feed volume and $C_{s_o} = C_F$, the following relation holds.

$$C_{s_o} V_{(liquid)} + C_{s_i} V^r_{liquid} = C_{s_o} V_m \qquad (3.7)$$

where V_m = effluent volume upto maximum concentration, assuming no forward mixing and complete equilibrium. Even though the forward mixing does occur and complete equilibrium is probably not attained, the equation is still a good approximation for the differential element of a break through curve.

Dividing by C_{s_o} and substituting $K_d = \dfrac{C_{s_i}}{C_{s_o}}$ in equation 3.7

$$V_{liquid} + K_d V^r_{liquid} = V_m \qquad (3.8)$$

It has been proposed by Martin and Synge (15) that a resin bed could be considered analogous to a packed distillation column and that the plate theory could be applied. Using this concept L.A. Matheson (16) suggested the following method for calculating the number of theoretical plates from experimental data.

$$P = \frac{2(C'+1)C'}{(W')^2} \qquad (3.9)$$

where P = number of theoretical plates

$$C' = \frac{\text{Amount of solute in resin phase}}{\text{Amount of solute in liquid phase}}$$
in equilibrium with resin phase

W' = Half width of elution curve an ordinate value of $1/e$ of the peak concentration (where e is the natural log base).

$$C' = \frac{C_{s_i} \, V^r_{liquid}}{C_{s_o} \, V_{liquid}} \tag{3.10}$$

From equation 3.6

$$C' = K_d \frac{V^r_{liquid}}{V_{liquid}} \tag{3.11}$$

Solving equation 3.8 for K_d and putting into equation 3.11

$$C' = \frac{(V_m - V_{liquid})}{V^r_{liquid}} \times \frac{V^r_{liquid}}{V_{liquid}} = \frac{(V_m - V_{liquid})}{V_{liquid}}$$

$$C' = \frac{(V_m - V_{liquid})}{V_{liquid}} \tag{3.12}$$

Substituting equation 3.12 into equation 3.9 and defining $W = W' \, V_{liquid}$;

$$P = \frac{2 \, V_m \, (V_m - V_{liquid})}{W^2} \tag{3.13}$$

where

W is measured in the same volume units as V^r, V_{liquid} and V_m.

Once the number of theoretical plates has been calculated, the H.E.T.P. can be determined by the relationship:

$$\text{H.E.T.P.} = h/P \tag{3.14}$$

where, h = height of the resin bed.

+++++++

CHAPTER IV

EXPERIMENTAL SET-UP

IV-1. Apparatus:

A schematic representation of the apparatus is given in Figure 5. The apparatus consists of (a) an ion-exclusion column (b) a constant temperature hot water both (c) a sample collection system and (d) a constant head device.

Ion-Exclusion Column

The column was made of pyrex glass of (0.6'' x 33.5'') and was jacketed for temperature control. It had a coarse sintered disc bottom for the resin supports. Resin bed temperature was maintained by circulating hot water through the jacket from the constant temperature hot water bath. The column was filled to a height of 24.5'' with sodium form of Dowex 50W-X8, 50-100 mesh. The flow rate was regulated by a stop cock at the bottom of the resin bed while maintaining a constant head. The dead volume (i.e. the volume below the sintered disc) was 12 ml.

Constant Hot Water Temperature Bath:

A Ganson's thermostated water bath was used to maintain the desired constant temperature of the resin bed. Hot water was circulated around the jacket by a circulating pump fitted with a regulator in circuit. The temperature inside the column could be maintained within $\pm 2^{\circ}C$.
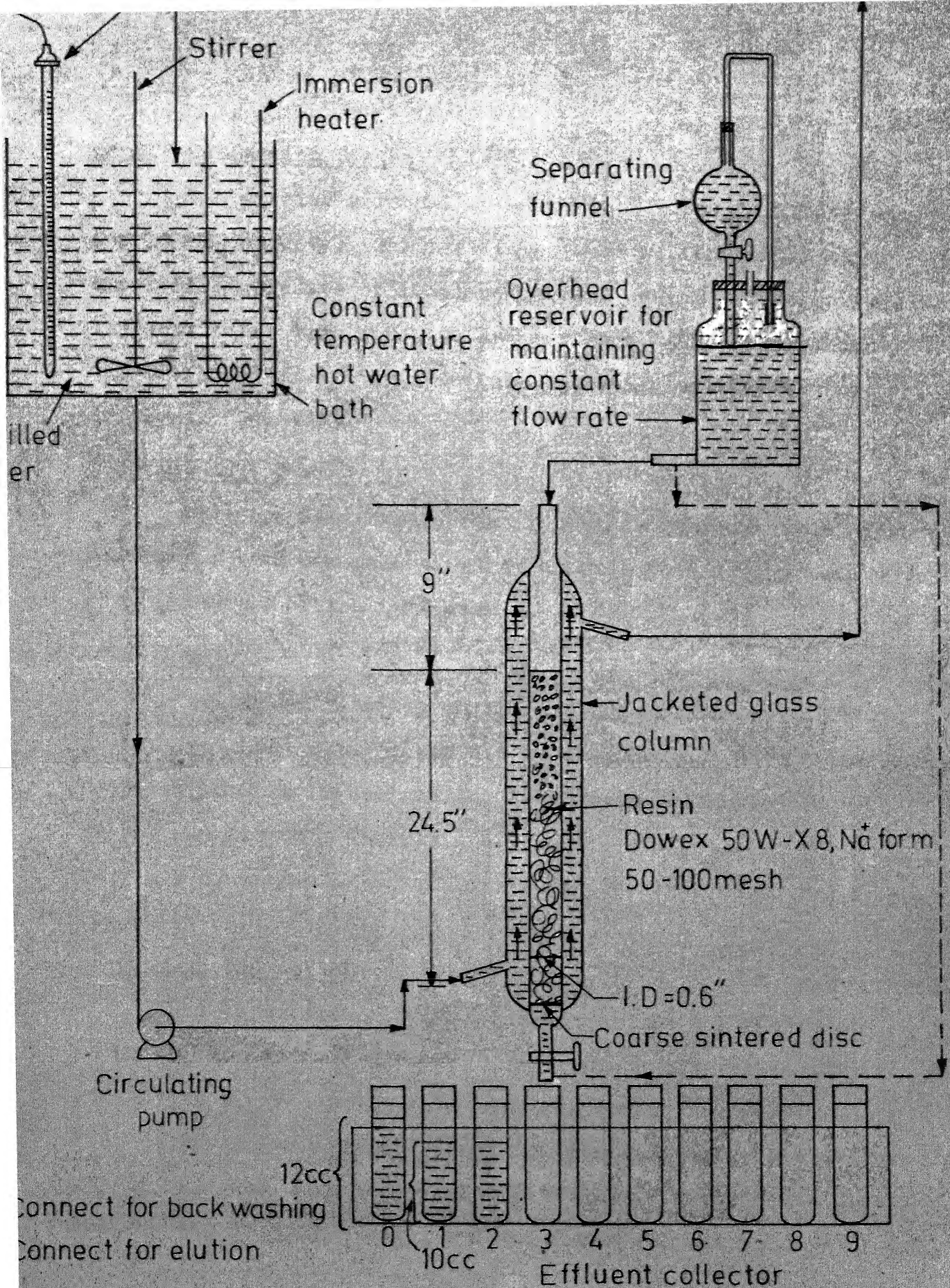
Fig. 5 - Experimental set-up.

Sample Collecting System:

The samples were collected in test tubes calibrated
for 10 ml capacity. The test tubes were calibrated by using
a certified pipette of 10 ml volume and making a fine mark.
The test tubes were labelled and numbered. The dead volume
was collected initially in a tube specially calibrated for
12 ml.

Constant Head Device:

The flow rate should remain constant throughout a run.
A constant head was maintained by an over head device made
of glass as described in Figure 5.

IV-2. Resin:

The strongly acidic cation exchange resin Dowex 50W-X8,
50-100 mesh, Na^+ form (marketted by Dow Chemical Co., U.S.A.)
was used in this investigation. The resin volume for 24.5"
height was 118 cm^3. The resin originally available in the H^+
form was converted into Na^+ form by a procedure described in
(Appendix D).

Setting-up the Column:

Requisite amount of ion-exchange resin was immersed
in water contained in a beaker for about 30 minutes to let it
swell. The slurry during this time was occasionally stirred.
Fine particles were decanted off and the residual resin slurry
was transferred into the column in stages. The colum was already
filled with distilled water and which was allowed to be displaced

by the incoming slurry. The resin filled column was tapped
gently to drive any trapped air bubbles. To ensure the
removal of entrained air bubbles or any remaining fine particles
and also to ensure even distribution of resin beads, the column
was 'backwashed' before use, i.e. a stream of distilled water
was run through the bed from the bottom at a flow rate
sufficient to loosen and suspend the exchange beads and
expand the bed by about 50-60 per cent. When the wash water
became clear, the flow of water was stopped and to resin
was allowed to settle in the column. The excess water was
drained off from the bottom. The water level was not allowed
to fall below the surface of the resin to obviate channelling
effect, which causes incomplete contact between the resin
and the surrounding solution.

IV-3. <u>Procedure</u>:

The column, after backwash, was allowed to stand so as
to let the height and temperature stabilize. Next the required
flow rate was adjusted by manipulating the stop cock at the
bottom. During this time the water level in the column was
taken slightly above the bed height and was maintained constant
at that level. The column was next checked for a vertical
position. It was thus ready for subsequent operations at the
desired temperature and flow rate.

Feed solution was made by dissolving requisite amount
of glycerol and sodium chloride in distilled water by weight

percentage. A known feed volume of this solution with specified concentration of sodium chloride and glycerol was run into a separating funnel. As soon as the water level just tipped of the resin bed, the stop cock of the separating funnel was open for the feed solution to run into the column. Simultaneously the effluent samples of 10 ml volume each were collected. As soon as the feed solution just touched the resin level eluent (distilled water) was fed to the column through constant head device maintaining the chosen flow rate. The samples were collected at the bottom continuously approximately 15-20 samples were taken depending upon the feed volume.

IV-4 Analysis:

The effluent samples collected in 10 ml fractions were subsequently analyzed for sodium chloride and glycerol content. The sodium chloride concentrations were determined by titrating a known small volume of the sample with silver nitrate (0.1N) using fluorescein as an indicator. The end point was determined by observing the change in color from white to slight pink. To get an acceptable value of end point three or four titration were performed.

The glycerol concentrations were determined by refractive index (n_d), and when salt was also present, by refractive index difference, because the refractive index of sodium chloride and glycerol were found to be additive. The density

of samples were determined by specific gravity bottle of
5 ml capacity. The calibration curve and model calculations
are given in (Appendix E and Appendix F) respectively.

+++++

CHAPTER V

RECYCLE PROCEDURE

V-1. Introduction

In the past, ion-exclusion had been carried out under conditions that would give an almost complete separation of the solutes in one pass through the ion-exchange resin bed. In this method the recovery of the desired product can be increased only at the expense of the average concentration. The product dilution was the chief disadvantage of the process.

A new method was developed by Simpson and Bauman (6) whereby the recovery can be improved without sacrificing the product concentration. In fact, it is often possible to obtain the product in concentrations greater than its initial concentration in the starting feed. This method is referred to here as the Recycle method.

V.2. Alternative Methods:

Depending on the manner in which we feed the effluent fractions back to the resin column in different two procedures can be followed:

1. Recycle method — A
2. Recycle method — B

Recycle Method – A:

This method is illustrated in the Figure 6. Essentially the effluent stream is continuously recycled back through a resin bed. For higher efficiency, the feed volume per cycle should be large enough so that the point of maximum cross-contamination in the effluent will be at or near the solute concentration of the feed solution. This may be accomplished in the laboratory by collecting the column effluents in small cuts throughout a run and then returning the cuts in the same order to the column for the following run and inserting a new feed at the point of maximum cross-contamination.

Once the individual waves have been identified as to shape and location, the effluent fractions could be divided into two groups. The first contains the major portion of the ionic solute and the second group contains most of the non-ionic solute. It is to be noted that first few effluents fractions would be discarded because they contain only water. These two groups and the new feed volume constituted the influent for the next cycle. This cycle could be repeated until the individual solute bands are broad enough to take product cuts which are equal to or in some cases higher in concentration than the original feed concentration.

Recycle Method – B:

This method is illustrated in Figure 7. In this method
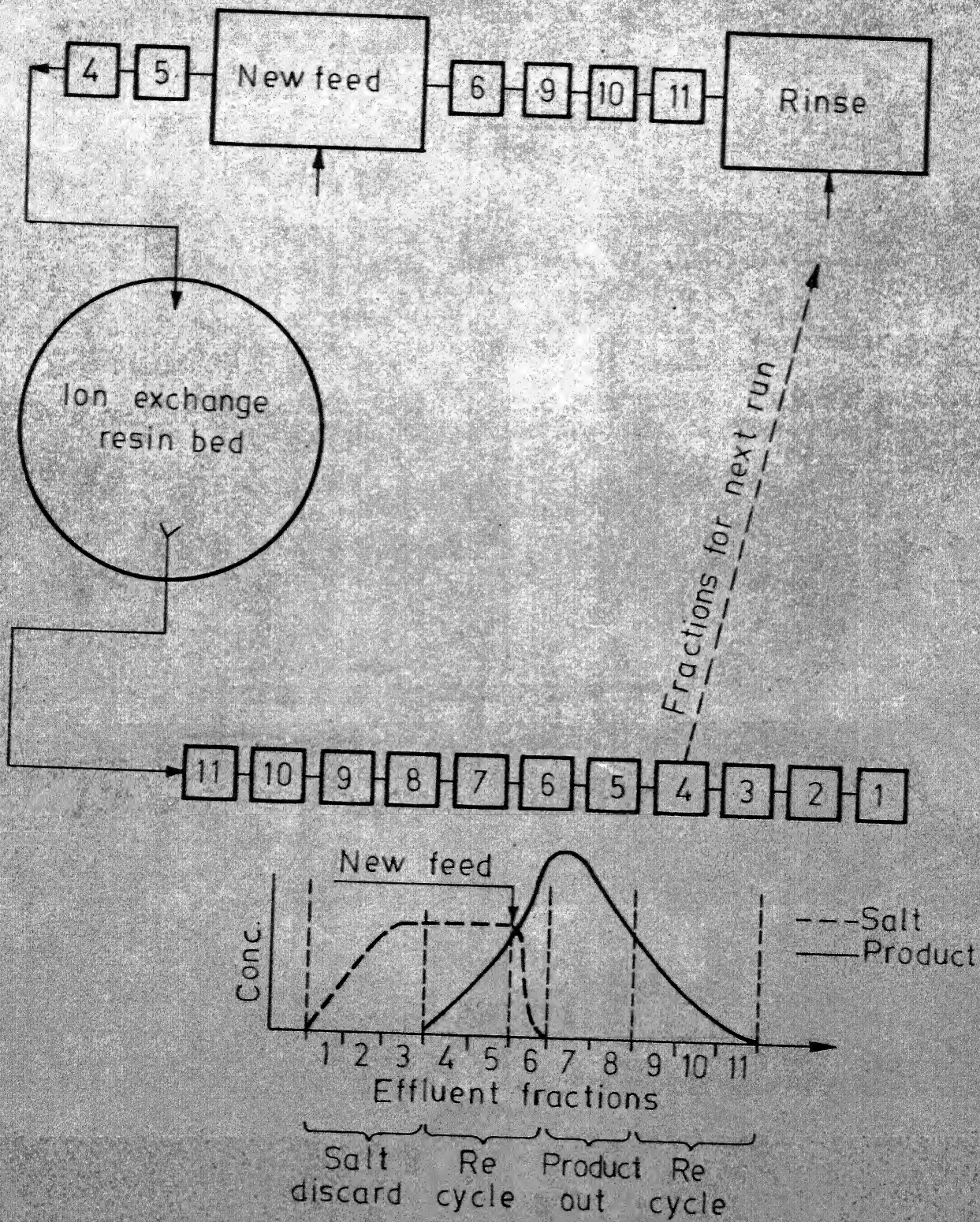
Fig. 6 - Diagrammatic sketch of recycle method for ion exclusion.
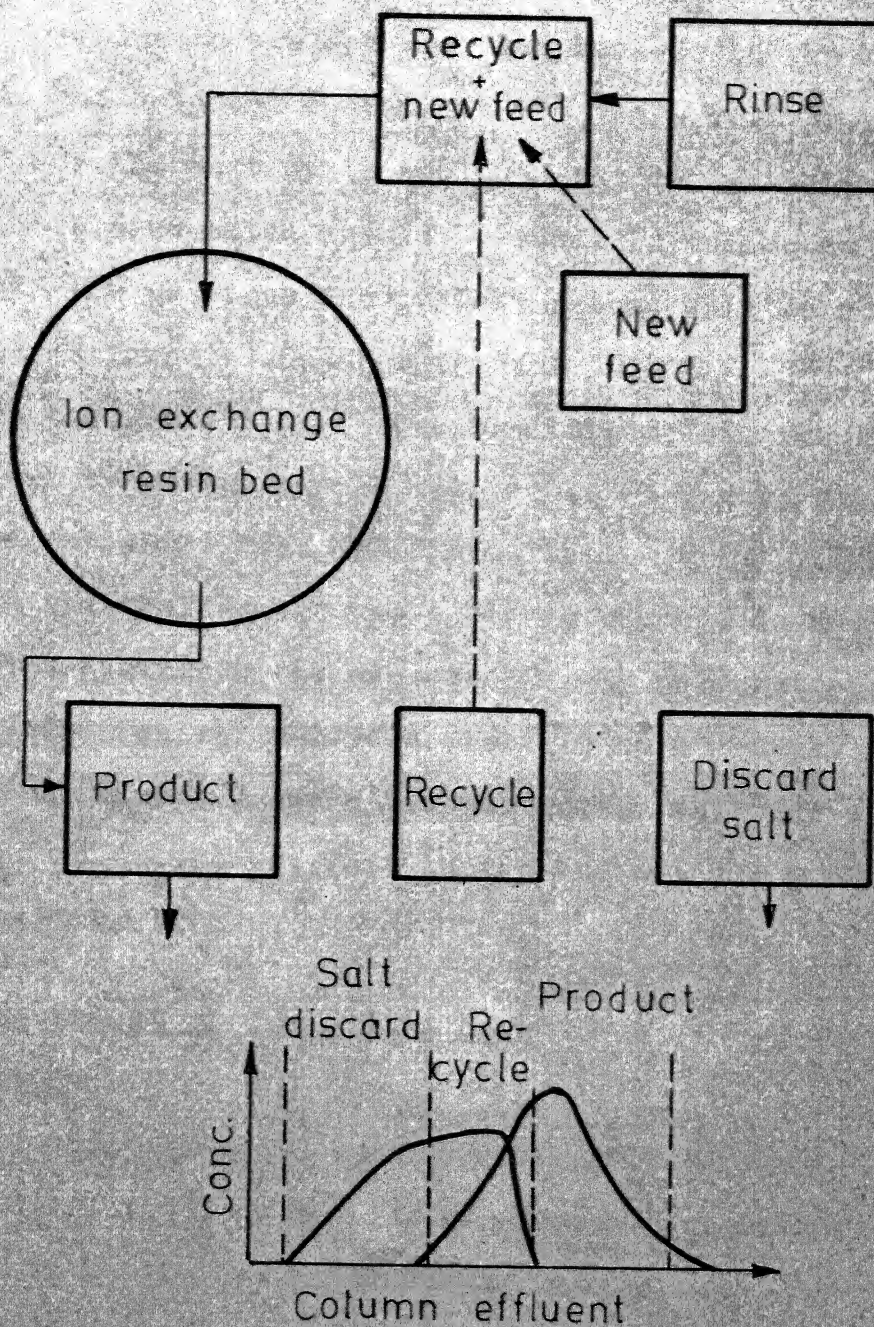
Fig. 7 - Diagrammatic sketch of batch recycle method for ion exclusion.

the total effluents are not recycled back as in the recycle method A, but only the contaminated part will be recycled. The feed for the next cycle would be the contaminated fraction plus the new feed which should be equal in volume and concentration to the previous feed. This method will be more suitable for large scale operation.

In the present work some preliminary studies were carried out by using recycle method A. The crude glycerol sample was obtained from M/S Hindustan Lever Ltd., Bombay. The crude glycerol sample was a highly viscous dark brown liquid, containing on the average 80 - 85 per cent weight glycerol. The ionized solute content is about 10 per cent weight. The ionized impurities were largely sodium chloride and small amounts of Na_2CO_3, soap, fatty acids and colouring matters were also present.

V-3. Pretreatment of Crude Glycerol:

[A] Dilution:

The crude glycerol sample was highly viscous, so the sample was diluted to about 30-35 per cent weight glycerol and 2-3 per cent weight sodium chloride approximately to avoid the excessive pressure drop across the resin bed.

[B] Filtration:

After dilution, filtration was carried out to remove the suspended solids, which choke the bed if not separated.

[C] Fats and Oil Removal:

The solution must be free from fats and oils, otherwise these tend to foul the exchange beads. So it was treated repeatedly with small amounts of CCl_4 and the organic layer removed each time.

[D] Color Removal:

Turbidity and colour materials in the solution must be kept to a minimum as these hinder and retard the exclusion process. So it was treated with activated charcoal to de-colorize the sample.

V-4. Procedure Adopted:

Sufficient quantity (about 40 ml) of feed volume was taken to create an area of cross contamination in the effluent. The effluent was collected in 10 ml fraction and each fraction was analyzed for sodium chloride and glycerol.

In the first run the shape and location of the individual solute waves were determined. The fractions were divided into two groups. The first group (A) contained the major portion of the ionic solute (sodium chloride) and the second group (B) contained most of the non-ionic solute (glycerol).

For first recycle, the first run was repeated but effluents fractions are not analyzed. The effluent was divided into two groups A and B as outlined above. The feed used for the first recycle was [A] + [40cc] + [B] in order the effluent of the first run was fed in the same order as it was collected.

Finally the bed was rinsed with distilled water. The effluent

was collected and analyzed for sodium chloride and glycerol.

The shape and location of the individual solute waves were

determined. Again the fraction wave divided into two group.

The operation was continued in a similar manner upto forth

recycle.

The cycle procedure is outlined on the following

page.

START

Feed=40cc

Exclu-
sion
Bed

Effluent were collected

| 0 | | 1 |...| 4 | | 5 |...| 8 | | 9 |...| 4 | | 5 |...| 20 |

Effluent were
analyzed and each
wave were identified

1.0 Feed

C_e/C_f

0

.32 .64   1.12

V_e/V_T

| 0 to 4 | 5 to 8 | 9 to 14 |

START

Feed=40cc

Exclu-
sion
Bed

Effluent were collected

| 0 | 1 |...| 4 | | 5 |...| 8 | | 9 |...| 4 | 1 | 16 | 17 |

Rejected   A   B   Rejected

| A | | 40cc | | B |

Exclu-
sion
Bed

Effluents were collected

| 0 | 1 |------| 25 |

Effluent were ana-
lyzed and each wave
were identified

1.0 Feed

C_e/C_f

0

.32 .96   1.76

V_e/V_T

CHAPTER VI

RESULTS AND DISCUSSION

Experimental data of the various runs are tabulated in Appendix G. The different variables which affect the ion-exclusion separation were (1) temperature, (2) flow rate,(3) feed volume, (4) resin cross-linkage, (5) resin particle size, (6) sodium chloride concentration and (7) glycerol concentration. Their effects are discussed in the beginning of this chapter. The effect of recycle on the separation was also studied. Correlations of the experimental variables as a function of H.E.T.P. have been made and, a discussion on the same is presented at the end of this chapter. The effect of only one variable was observed at a time while others remained constant.

VI-1 Effect of Temperature:

In the present investigation the effect of temperature has been studied in the range 20-80°C. The experimental data are given in Table 10 and a few elution curves are shown in Figure 8. It was observed that at low temperature, the trailing edge of the sodium chloride and glycerol wave were not sharp and the former extended across the heart of glycerol wave, thus contaminating and diluting the desired product. Increasing the temperature gave much sharper waves and also improved the concentration of the desired product. It was also observed

Operating conditions:

(1) Feed volume = 30cc (0.25 V_T)

(2) Resin = Dowex 50W-X8, Na^+ form

(3) Flow rate = 25cc/min (0.337 gal/ ft^2/min)

(4) Size = 50 - 100 mesh

(5) Feed conc^n: Glycerol = 33.75% W

    and sodium chloride

    = 2.50% W

V_T = 118 cm^3 (0.0040 ft^3)

○ Temp. 20°C
● Temp. 65°C
△ Temp. 80°C

Glycerol

Sodium chloride

Ce/Cf

Ve/V_T
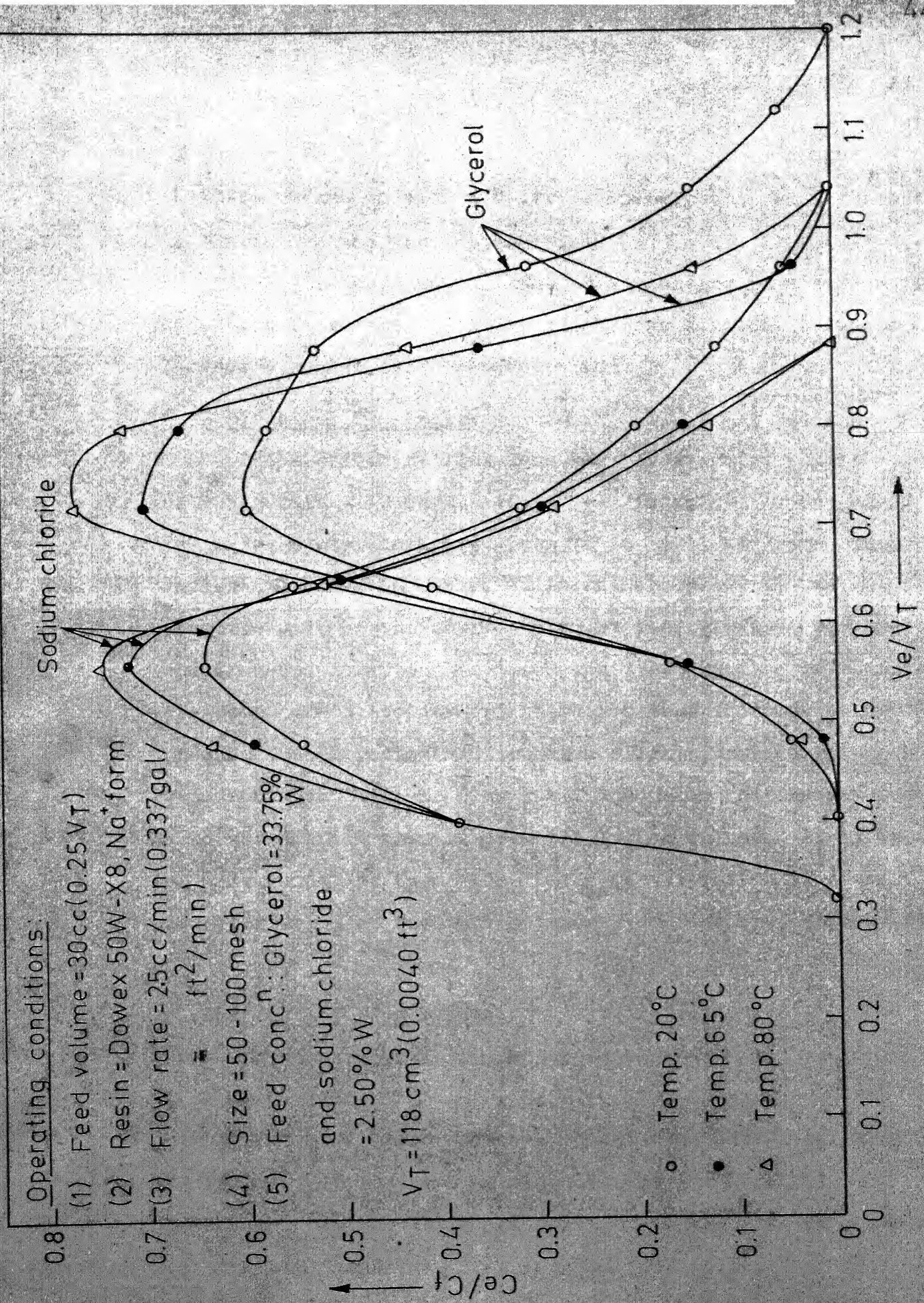
Fig 8 - Effect of temperature.

that the maxima of sodium chloride and glycerol with respect to elution volume were not shifted.

It is believed (7) that this trailing is a viscosity effect, because the viscosity of aqueous glycerol solution increases rapidly with concentration.

VI-2 Effect of Flow Rate:

The effect of flow rate was investigated in the range of 2.5 cc/min - 10 cc/min (0.337 gal/ft^2/min - 1.350 gal/ft^2/min). The experimental data are given in Table 11 and some elution curves are shown in Figure 9. It was observed that at low flow rate the wave of the desired product i.e. glycerol was sharp and the concentration was also high. As the flow rate increased the wave turned broader and there was thus decrease in the product concentration. There is a slight increase in contamination with increase flow rate. The maxima of the waves were also not shifted with respect to elution volume. It is reasonable to assume that at lower flow rates the operation is nearer to equilibrium, good separation takes place and product concentration is higher.

VI-3 Effect of Feed Volume:

The effect of feed volume was studied in the range of 5 to 40 cc (0.042 V_T to 0.336 V_T). The experimental data are given in Table 12 and some typical elution curves are shown in Figure 10. It was observed that smaller feed volumes, results in smaller contamination and dilution of the desired product but the separation was good. As the feed volume was increased,

Operating conditions:
(1) Temperature = 80°C
(2) Feed volume = 30cc (0.25V_T)
(3) Resin = Dowex 50W-X8, Na$^+$ form
(4) Size = 50 - 100 mesh
(5) Feed concentration: Glycerol = 33.75%W and sodium chloride = 2.50%W
$V_T = 118$ cm^3 (0.0040 ft^3)

○ 10cc/min (1.350 gal/ft^2/min)
● 7.5cc/min (1.029 gal/ft^2/min)
△ 5.0cc/min (0.675 gal/ft^2/min)
▲ 3.5cc/min (0.4725 gal/ft^2/min)

Sodium chloride   Glycerol

C_e/C_f

V_e/V_T

Fig. 9 - Effect of flow rate.

Fig. 10 - Effect of feed volume.

Operating conditions:

(1) Temperature = 80°C

(2) Flow rate = 3.70 cc/min (.0499 gal/ft²/min)

(3) Resin = Dowex 50W-X8, Na⁺ form

(4) Size = 50-100 mesh

(5) Feed concentration: Glycerol = 33.75% W & NaCl = 2.50% W

$V_T = 118 \text{ cm}^3 (0.0040 \text{ ft}^3)$

● 5cc (0.042 V_T)

△ 7cc (0.059 V_T)

▲ 10cc (0.084 V_T)

○ 15cc (0.126 V_T)

Ve/VT

Ce/Cf

Glycerol

Sodium chloride

glycerol concentration increased but the contamination was also greater. It was also observed that the maxima of the elution curve were shifted with respect to the effluent volume in the forward direction and the curves tended to widen. The maximum feed volume may not be greater than about 0.50 V_T of the resin bed volume, because the separating capacity of the resin bed would be exceeded.

VI-4 Effect of Cross-Linkage:

A few separations were tried with Dowex 50, Na^+ form in different cross-linkages (4, 8 and 10 per cent). The experimental data are given in Table 13 and the elution curves are shown in Figure 11. It was observed that at lower cross-linkage, the elution curve peaks shifted in the forward direction, more so for glycerol. This is due to fact that V^r is an inverse function of cross-linkage (17,18). The separation improved but the concentration decreased. The curve were also not sharp. Conversely, as the cross-linkage increased the product concentration increased and the elution curve also sharpened, but separations were poor. The V_{liquid} was also found to be function of cross-linkage as given in Appendix C.

It is believed that separation were poor at a higher cross-linkage because as the cross-linkage increased, the porosity decreased, so, the diffusion would be slow and separation would also be poor.

Operating condition:

(1) Temperature = 80°C
(2) Feed volume = 30cc (0.25 V_T)
(3) Flow rate = 25cc/min (0.337 gal/ft^2/min)
(4) Resin = Dowex 50W, Na$^+$ form
(5) Size = 20-50 mesh
(6) Feed conc^n. glycerol = 34.6%W and NaCl = 2.60%W

V_T = 118 cm^3 (0.0040 ft^3)

○   X-4
●   X-8
△   X-10

Glycerol

Sodium chloride

C_e/C_f

V_e/V_T
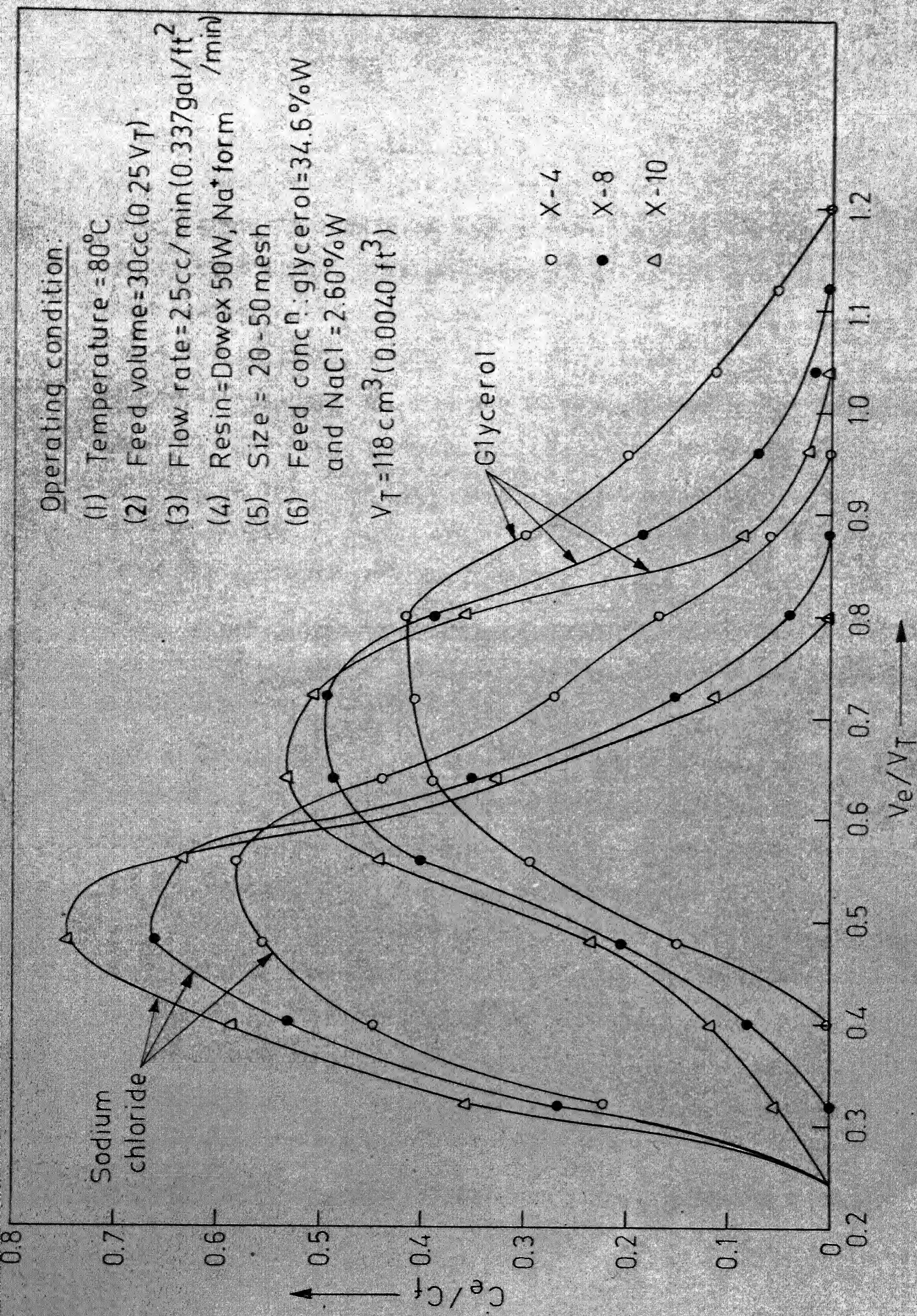
Fig 11 – Effect of cross linkage.

VI-5 <u>Effect of Particle Size:</u>

The effect of particle/could be studied for 50-100,

size

20-50 mesh sizes only because of non-availability of the

other mesh sizes. The experimental data, are given in Table 14

and elution curves are shown in Figure 12. It was observed

that for smaller particle size, the curves were sharp, the

separation was found to be better and the peak concentrations were

higher. For small particle size the rate of equilibrium

attainment was shown to be faster for ethylene glycel by

Bauman and Wheaton (4). The same may be expected to hold

good for glycerol or other non-ionic solutes.

VI-6 <u>Effect of Sodium Chloride Concentration:</u>

The effect of ionic concentration (NaCl) were investigated

in the range of (1.260/W to 16.950°/. W). The experimental data

are given in Table 15(A) and Table 15(B) for two separate feed

volumes. A few elution curves are shown in Figure 13(a) and

13(b) for corresponding feed volumes. It was observed that

at low ionic concentration the separation was good and

contamination was low but the product concentration was also

low. As the concentration was increased the elution curve

shifted in the forward direction. The peak glycerol concen-

tration found to increase as the ionic concentration was

increased, but the separations were slightly poor because of

larger contaminated zones. The peak NaCl concentration remained

more or less the same. It has been shown by Shurts (10) that

Operating conditions:

(1) Temperature = 80°C
(2) Feed volume = 30 cc (0.25 V_T)
(3) Flow rate = 2.5 cc/min (0.337 gal/ft²/min)
(4) Resin = Dowex 50W-X8, Na⁺ form
(5) Feed conc'n, glycerol = 34.6% W and NaCl = 2.6% W
V_T = 118 cm³ (0.0040 ft³)

● 50 - 100 mesh
○ 20 - 50 mesh
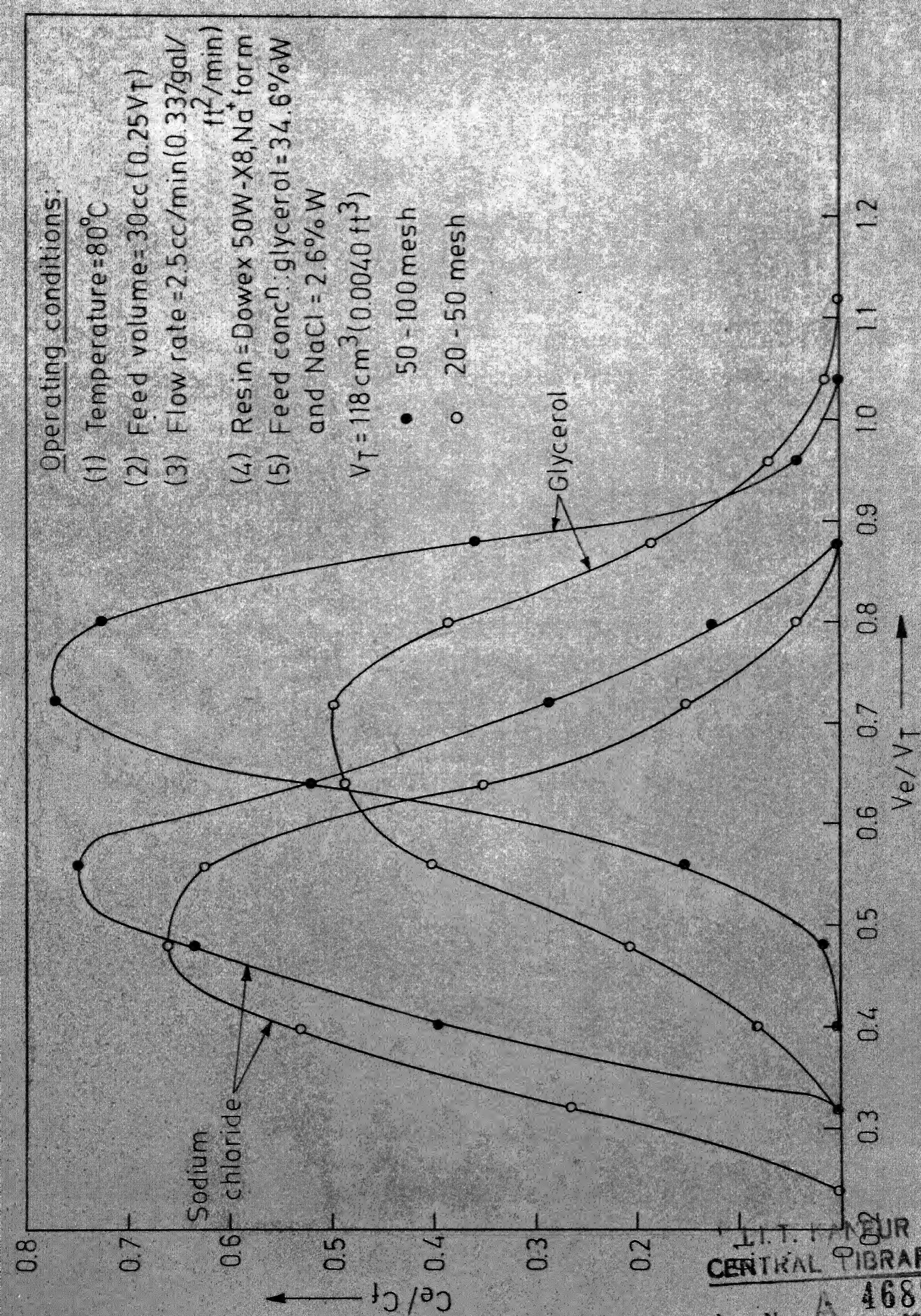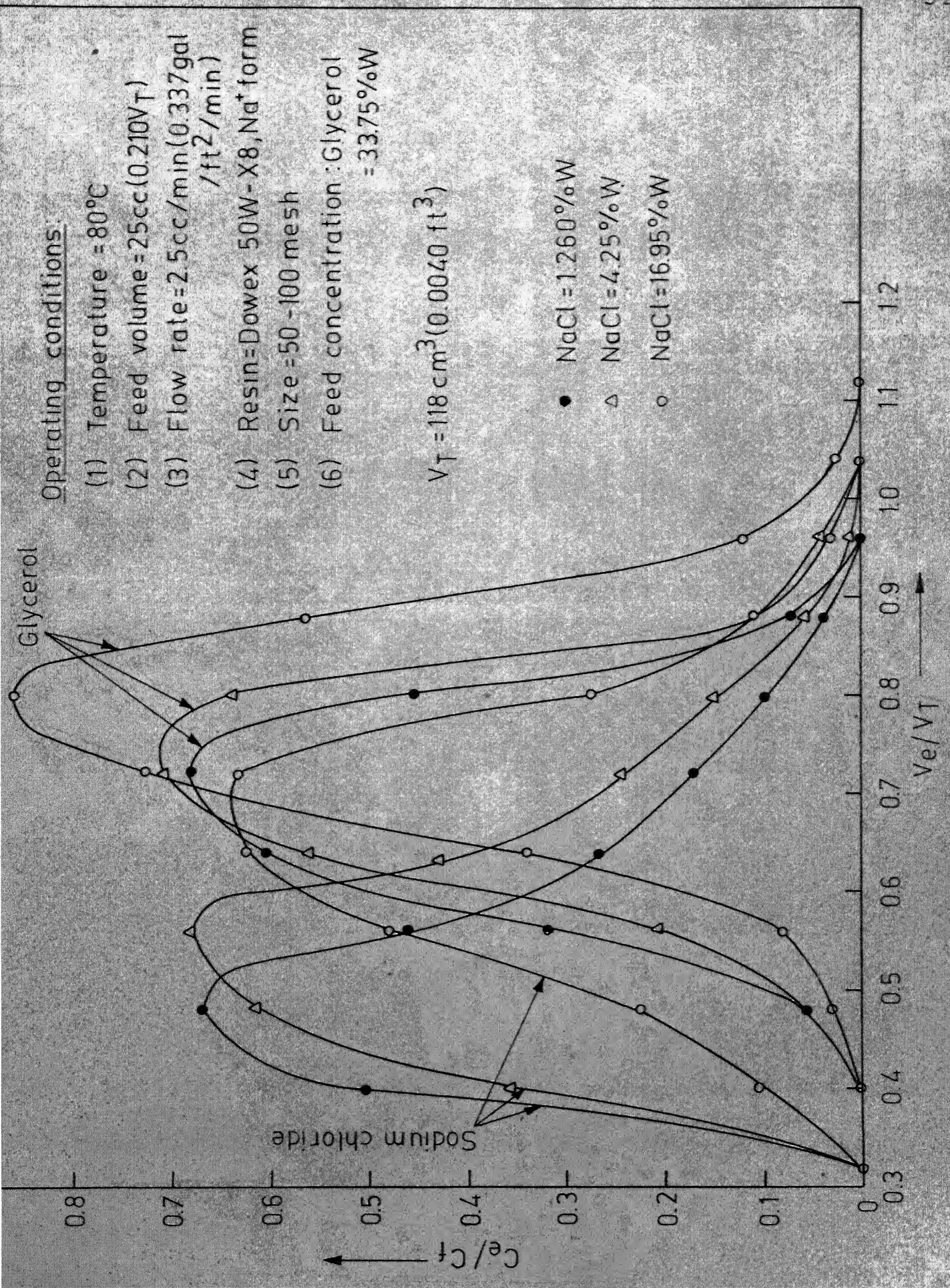
Glycerol

Sodium chloride

Ve/V_T →

Ce/Cf →

Fig. 12 - Effect of particle size.

Operating conditions:

(1) Temperature = 80°C
(2) Feed volume = 25cc (0.210V_T)
(3) Flow rate = 2.5cc/min (0.337 gal /ft²/min)
(4) Resin = Dowex 50W-X8, Na⁺form
(5) Size = 50-100 mesh
(6) Feed concentration : Glycerol = 33.75%W

V_T = 118 cm³ (0.0040 ft³)

● NaCl = 12.60%W
△ NaCl = 4.25%W
○ NaCl = 16.95%W

Glycerol

Sodium chloride

C_e/C_f

V_e/V_T →

Fig 13(a)- Effect of sodium chloride concentration

Operating conditions:

(1) Temperature = 80°C

(2) Feed volume = 10cc(0.084 V_T)

(3) Flow rate = 2.5cc/min(0.337gal /ft²/min)

(4) Resin = Dowex 50W-X8, Na⁺-form

(5) Size = 50-100 mesh

(6) Feed concentration: Glycerol =33.75%W

V_T = 118 cm³(0.0040ft³)

● NaCl ≈12%W

○ NaCl =7.85%W

△ NaCl=1.260%W

Glycerol

Sodium chloride

V_e/V_T ⟶

⟵ C_e/C_f
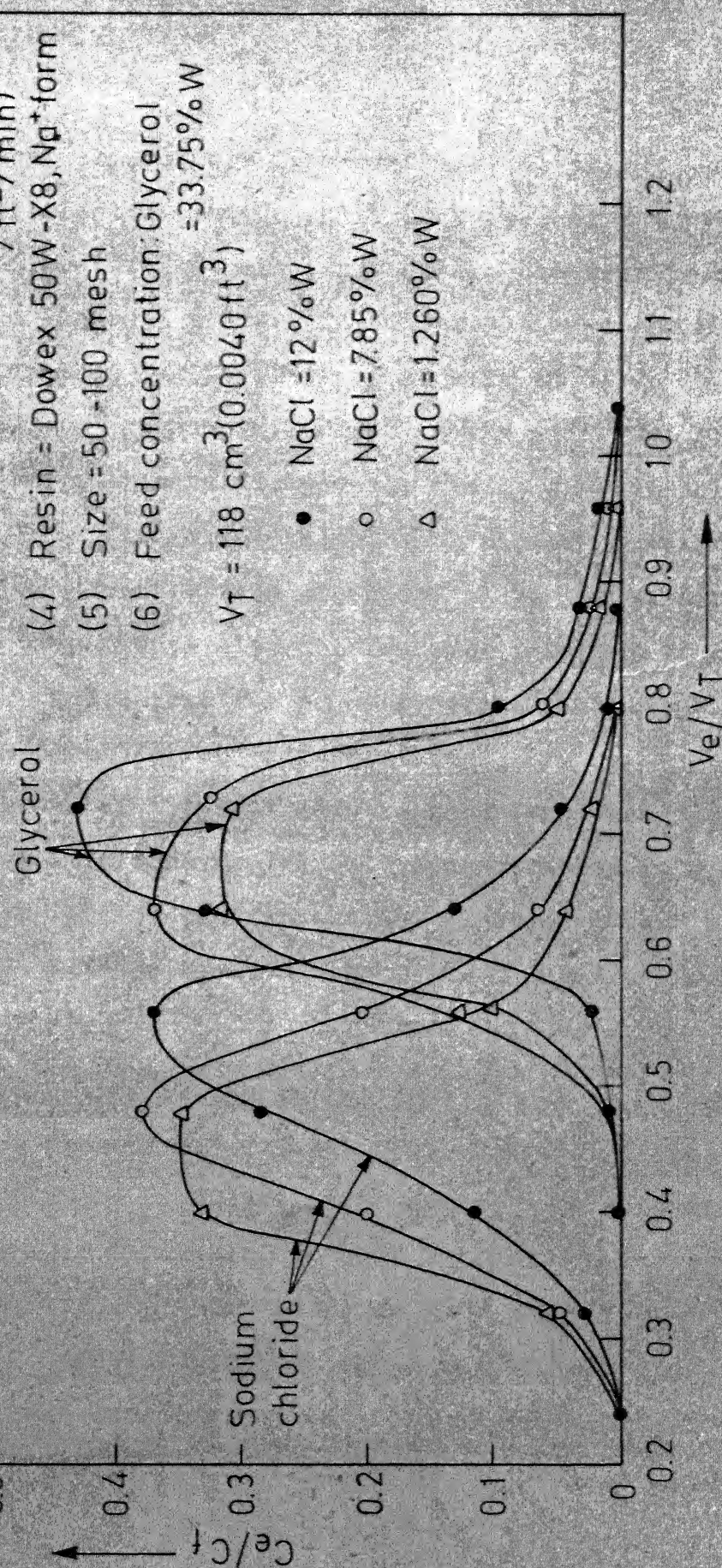
Fig.13(b)-Effect of sodium chloride concentration

as the concentrations were increased the not only   distribution coefficient of sodium chloride increased but glycerol distribution coefficient also increased. The results of Shurts are shown in Figure 3 and 4. The distribution coefficient of glycerol increases due to presence of salt (NaCl). This delays the appearance of glycerol fraction in the different stream. However as the salt wave disappears, the K_d of glycerol rapidly falls to its normal value, thus causing a large amount of glycerol to transfer from the internal to the external phase. This sudden transfer of glycerol from the internal to external liquid phase causes glycerol to concentrate at this point more or less equal to feed concentration.

The shape of the salt wave is dependent upon the initial concentration. Increasing the salt concentration, increased the salt distribution coefficient and also dehydrated the resin. Both of these factors also tend to sharpen the trail of the salt wave.

VI-7 Effect of Glycerol Concentration:

In the present investigation the glycerol concentration ranged from 6.75 to 51.40 per cent weight. The experimental data are given in the Table 16 and a few elution curves are shown in Figure 14. It was observed that the effect of glycerol concentration was negligible on the degree of separation at low concentration. Even though for higher glycerol concentration the distribution coefficient of the salt increased

Fig. 14 - Effect of glycerol concentration.

Operating conditions:
(1) Temperature = 80°C
(2) Feed volume = 30 cc (0.25 V_T)
(3) Flow rate = 2.5 cc/min (0.337 gal/ft^2/min)
(4) Resin = Dowex 50W-X 8, Na$^+$ form
(5) Size = 50-100 mesh
(6) Feed conc^n: Sodium chloride = 2.50 %W

V_T = 118 cm^3 (0.00040 ft^3)

○ 10.625 % Glycerol
● 16.250 % Glycerol
△ 51.40 %

Sodium chloride
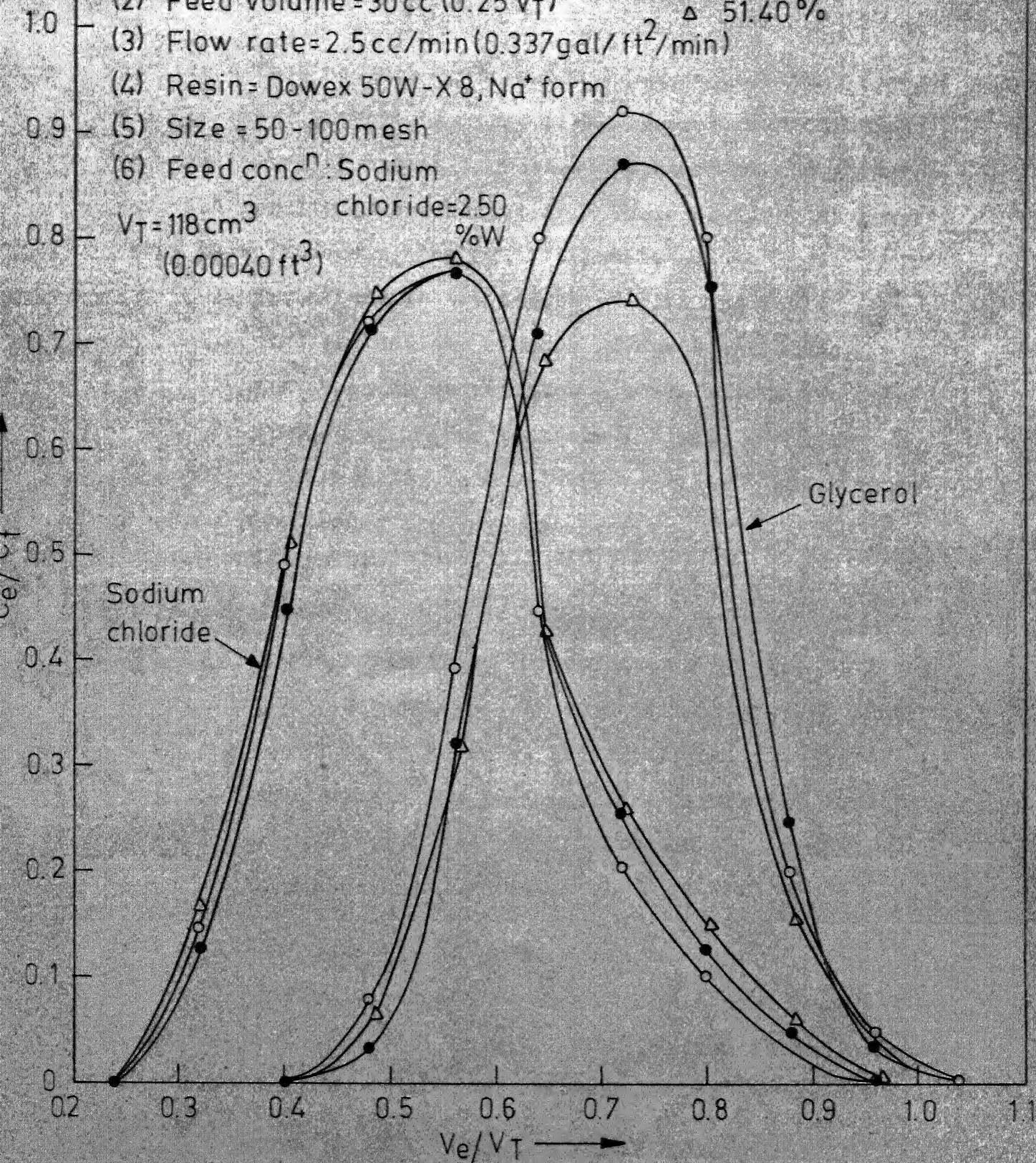
Glycerol

but the affect appears to be swamped out, in an actual operation by all the other factors. At very high glycerol concentration the trail wave of the salt crosses the heart of the glycerol wave and results in contamination. There could be at least three factors causing the salt to trail:

1. Variations in the degree of trailing would suggest that the viscous glycerol solutions may cause an uneven flow pattern within the column.

2. Viscous solutions decrease the rate of diffusion which results in a greater volume of cross-contaminated effluent material.

3. Higher glycerol concentration decreased the activity of both the salt and sulfonic acid group. A decrease in the activity of the salt tends to make it less ionic in character, thus contributing to an increased K_d value. A decrease in activity of the ionic group within the resin would decrease the ion-exclusion properties of the resin and would also tend to increase the K_d of salt.

VI-8 Effect of Recycle:

For study of recycle a sample was procured from M/S Hindustan Lever Limited, Bombay. Its chemical nature has been described earlier. The effect of recycle was investigated upto the fourth cycles. The experimental data are given in Table 17

and the elution curves of I recycle and for IV recycle are shown in Figure 15 and Figure 16 respectively. It was observed that as the number of recycles increased the product concentration and the separation improved. The recycle procedure described earlier made it possible to obtain product cuts at the same or even higher concentration than the feed concentration as shown in Figure 17 and 18. An important observation from the industrial point of view is that the color bodies were also eluted along with the salt fractions leaving an almost colorless glycerol fraction.

As stated above the experiments on recycle were carried out with crude glycerol and no attempt was made to change the salt concentration of the sample. It is possible to improve the product concentration by the addition of the sodium chloride to the commercial glycerol sample in conjunction with a recycle procedure. This can become a subject for further investigation of the recycle procedure.

VI-9 <u>Correlation of Variables</u>:

The expressions,

$$P = \frac{2 V_m (V_m - V_{liquid})}{W^2} \tag{6.1}$$

$$H.E.T.P. = h/p \tag{6.2}$$

(Refer to Eqns. 3.13 and 3.14 in Chapter III)

make it possible to calculate the number of theoretical plates

Fig.15- Elution curve after I recycle

Operating conditions:

(1) Temperature = 80°C
(2) Feed volume = 40 cc (339 V_T)
(3) Flow rate = 2.5 cc/min (34 g/ft²/min)
(4) Resin = Dowex 50W-X8
(5) Mesh = 50-100 mesh
(6) Feed concⁿ: Glycerol = 33% W &
    NaCl = 2.86% W

Feed for II recycle

5 to 12 · 40cc
13 to 21

Feed for I recycle

5 · 6 · 7 · 8 · 40cc · 9 to 14

Product

Glycerol

Feed

Sodium chloride

V_e/V_T ——

C_e/C_t

Operating conditions:

(1) Temperature = 80°C

(2) Feed volume = 40cc(.339V_T)

(3) Flow rate = 2.5cc/min(3/4g/ft²/min)

(4) Resin = Dowex 50W-X8, Na⁺ form

(5) Size = 50-100 mesh

(6) Feed concentration: Glycerol = 33%W, NaCl = 2.86%W

Feed for IV recycle

| 9 to 16 | + | 40cc | + | 17 to 34 |

Glycerol

Sodium chloride

← Waste →   ← Recycle →   ← Product →

C_e/C_f
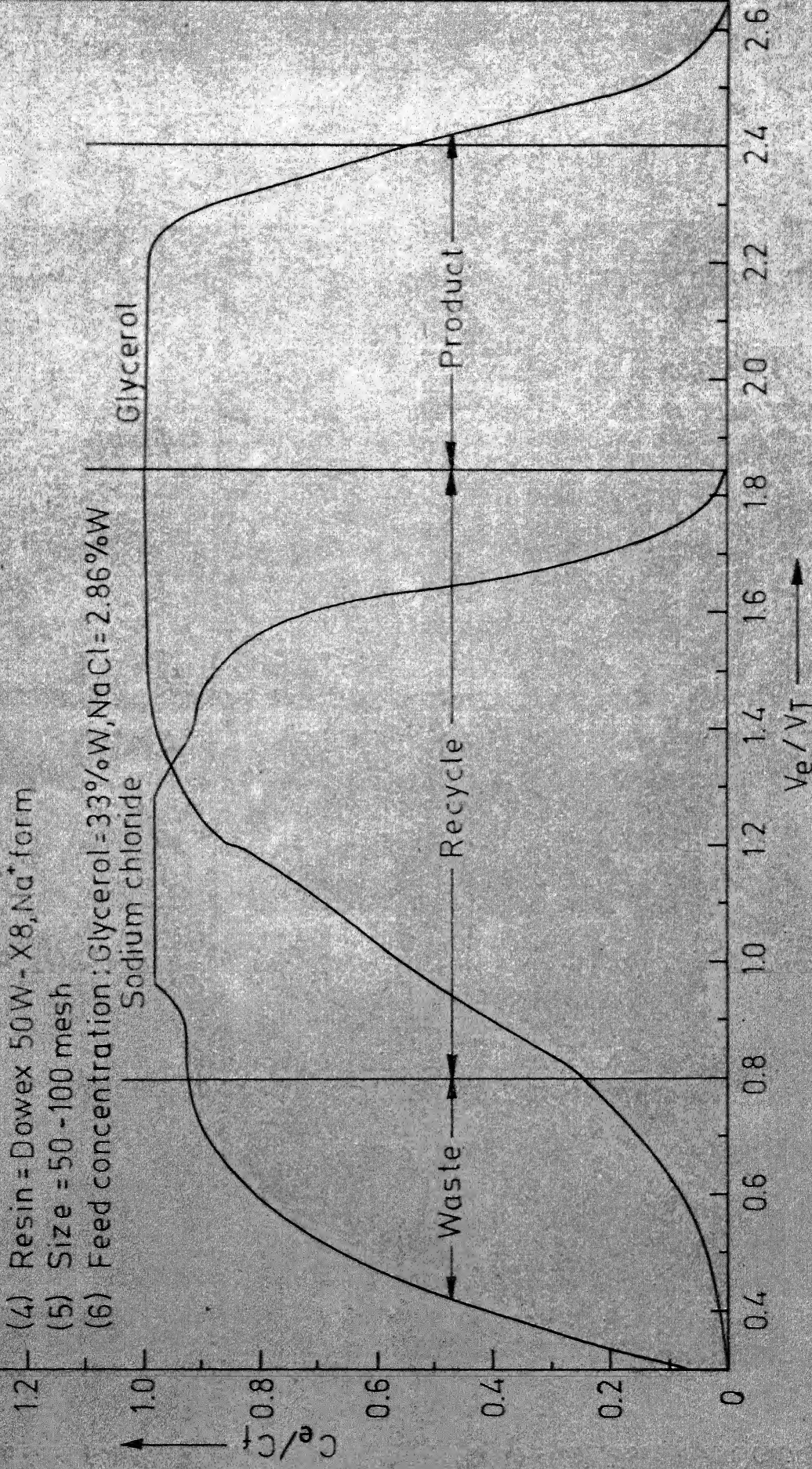
Fig.16 - Elution curve after IV recycle

V_e/V_T →

Operating conditions:
(1) Temperature = 80°C
(2) Feed volume = 40cc (0.336 V_T)
(3) Flow rate = 2.5 cc/min (0.337 gal/ft^2/min)
(4) Resin = Dowex 50 W-X8, Na$^+$ form
(5) Size = 50-100 mesh
(6) Feed concentration: Glycerol = 33%W
    and Sodium chloride = 2.86%W

○ Without recycle
● First recycle
△ Second recycle
▲ Third recycle
□ Fourth recycle

V_T = 118 cm^3 (0.0040 ft^3)

C_e/C_f

V_e/V_T →
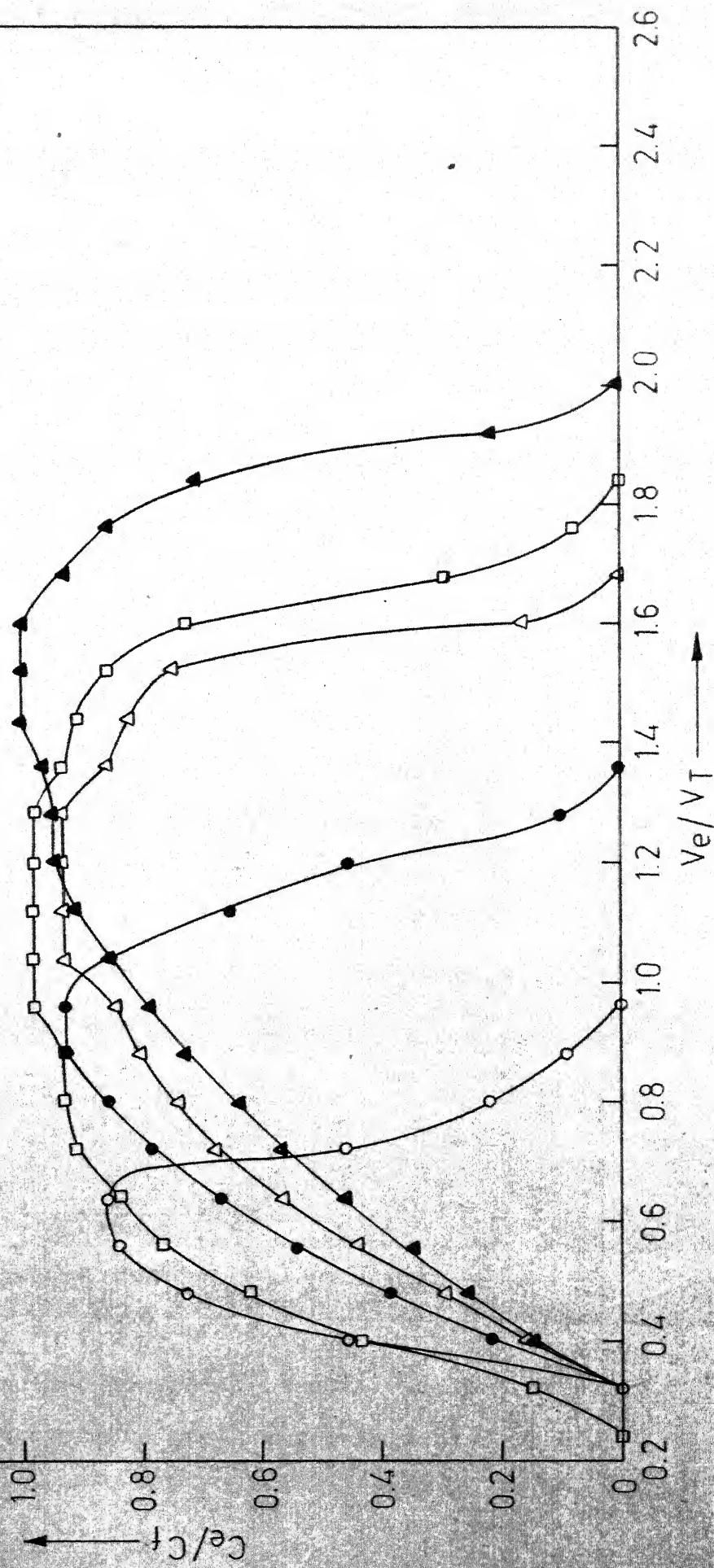
Fig. 18 - Effect of recycle on sodium chloride.

and in turn to determine the H.E.T.P. It may be noted that the mathematical model incorporated in the expression 6.1 is yet not conclusive. Any alternatives in terms of more accurate expressions have not yet been reported. Bauman and Simpson (5) have drawn correlations, based on this model of H.E.T.P. with operating variables. Since the present study was not directed at the mathematical analysis of the column, the above expression for H.E.T.P. has been assumed and the resulting correlations have been compared with the only available results for ethylene glycol.

Temperature:

It was observed that the maxima of elution curves of glycerol did not change with respect to elution volume i.e. V_m remained constant. The interstitial volume (V_{liquid}) also remained constant for a particular resin (it being a function of particle size and cross-linkage). Therefore, with V_m and V_{liquid} remaining constant,

$$P = \text{Constant} \times \frac{1}{W^2} \qquad (6.3)$$

or

$$\text{H.E.T.P.} = \frac{h}{P} = \frac{h}{\text{Constant}} W^2 \qquad (6.4)$$

For a given bed height

$$\text{H.E.T.P.} \propto W^2$$

From the elution curves it is observed that with increasing temperatures the width (i.e. W) decreases as

tabulated in Table 2. A log-log plot of temperature versus W^2 shows a linear variation. Therefore, H.E.T.P. should also vary linearly with temperature on log-log plot as shown in the final form in Figure 19. The slope of the curve was found to be close to 0.5. As the temperature increased from 20° to $80^\circ C$, the H.E.T.P. were found to decrease from 2.257 to 1.051 inches.

The diffusivity of glycerol in Dowex 50 W -X8 has been measured over a range of temperature by Tayyabkhan and White [19] and has been shown to increase with temperature. The increase in diffusivity should be reflected in the decrease of H.E.T.P. would have been found to be the case.

Flow Rate:

It can be seen that V_m of elution curves with respect to flow rates remained invariant. On a similar reasoning as given in the effect of temperature, H.E.T.P. here would also be proportional to W^2. However, unlike the previous one the W^2 increases linearly with flow rate as shown in Table 3. Therefore, H.E.T.P. should also increase in a linear manner with flow rate on a log-log plot as shown in Figure 20. The slope of the curve was found to be 0.5. Simpson and Bauman [5] also reported similar dependence for ethylene glycol. It shows that H.E.T.P. is directly proportional to the square root of the flow rate. The following relation is proposed for H.E.T.P. variation with flow rate.

TABLE 2

EFFECT OF TEMPERATURE ON H.E.T.P.

S. No.	Temp., $^\circ C$	V_m x118 cc	Peak Glycerol Concn.	W x 118 cc	W^2x118^2 cc^2	P	H.E.T.P. inches
1	20	0.72	0.60	0.2125	0.0452	10.855	2.257
2	30	0.72	0.63	0.1925	0.0371	13.232	1.851
3	40	0.72	0.66	0.1825	0.0333	14.702	1.666
4	50	0.72	0.68	0.1750	0.0306	16.000	1.531
5	58	0.72	0.69	0.1675	0.0281	17.485	1.401
6	65	0.72	0.70	0.1550	0.0240	20.400	1.200
7	75	0.72	0.72	0.1475	0.0218	22.562	1.085
8	80	0.72	0.77	0.1450	0.0210	23.331	1.051

TABLE 3

EFFECT OF FLOW RATE ON H.E.T.P.

S. No.	Flow Rate gal/ft^2/min	V_m x118 cc	PeakGlycerol conc.	Wx118 cc	W^2x118^2 cc^2	P	H.E.T.P. inches
1	0.337	0.80	0.770	0.1450	0.0210	29.8	0.770
2	0.472	0.80	0.750	0.1525	0.0233	28.8	0.850
3	0.540	0.80	0.730	0.1575	0.0248	27.1	0.904
4	0.600	0.80	0.720	0.1675	0.0281	23.9	1.022
5	0.675	0.80	0.700	0.1750	0.0306	21.9	1.116
6	0.810	0.80	0.695	0.1775	0.0315	21.3	1.150
7	1.012	0.80	0.665	0.1875	0.0352	19.1	1.281
8	1.350	0.80	0.645	0.1900	0.0361	18.6	1.317
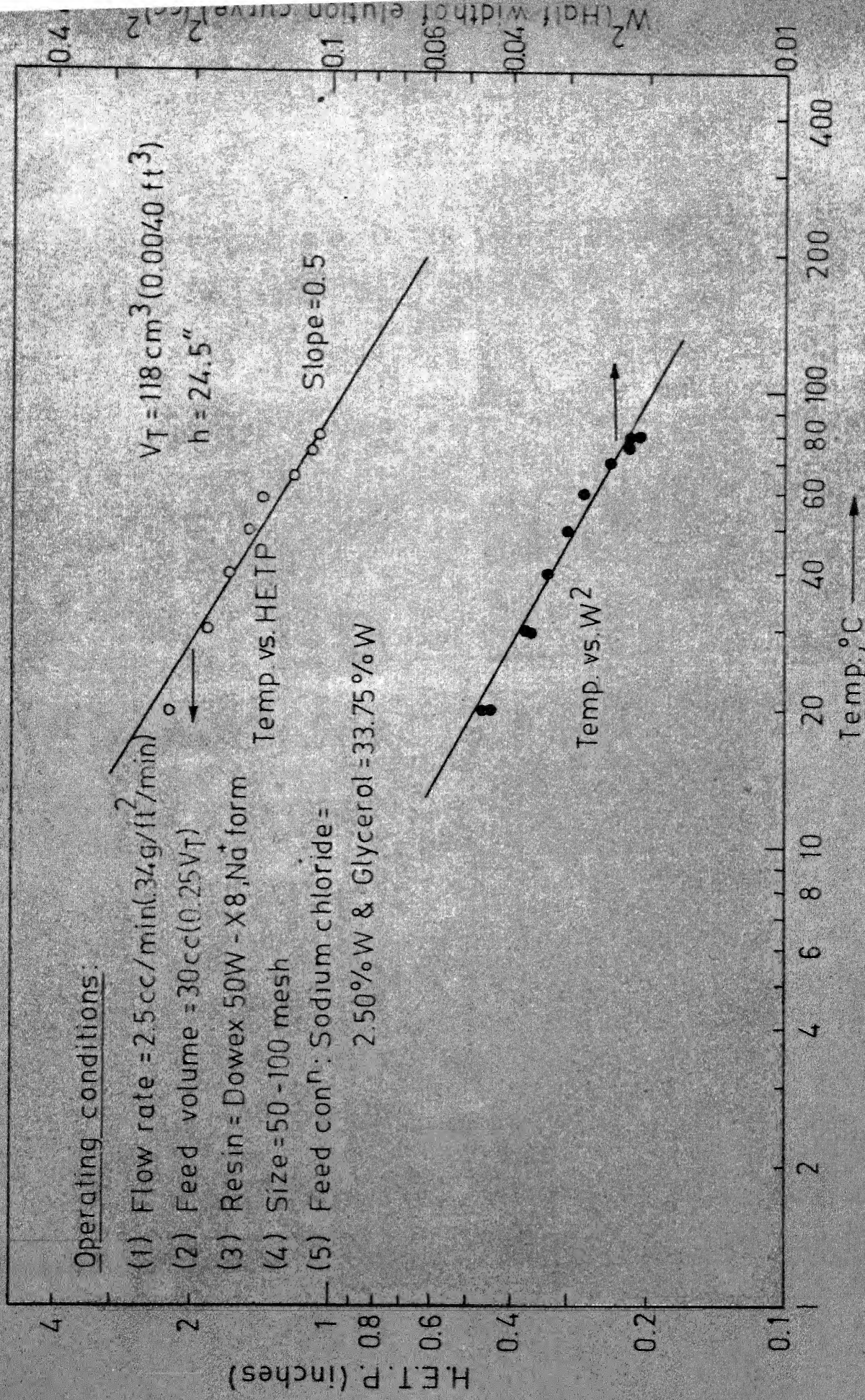
Fig 19—Effect of temperature on HETP elution of glycerol

Operating conditions:

(1) Flow rate = 2.5 cc/min (34 g/ft²/min)

(2) Feed volume = 30 cc (0.25 V_T)

(3) Resin = Dowex 50W - X8, Na⁺ form

(4) Size = 50 - 100 mesh

(5) Feed conⁿ: Sodium chloride =

2.50% W & Glycerol = 33.75 % W

V_T = 118 cm³ (0.0040 ft³)

h = 24.5"

Slope = 0.5

Temp. vs. HETP

Temp. vs. W²

Fig. 20 - Effect of flow rate on H.E.T.P.

Operating conditions:

(1) Temperature = 80°C

(2) Feed volume = 30cc (.25 V_T)

(3) Resin = Dowex 50W-X8, Na^+ form

(4) Size = 50 - 100 mesh

(5) Feed concentration : Sodium chloride = 2.50% W
   and Glycerol = 33.75% W

(Total bed volume) V_T = 118 cm^3 (.0040ft^3)

(Resin bed height) h = 24.5"

Flow rate vs. H.E.T.P.

Flow rate vs. W^2

Slope = 1/2

W^2 (Half width of elution curve), cc^2

H.E.T.P. (inches)

Flow rate in gal./ft^2/min

$$H.E.T.P = K \ (\text{flow rate})^{\frac{1}{2}}$$

Where K depends upon the column characteristics, operating conditions and the system considered. As the flow rates increased from 2.5 cc/min(0.337 gal/ft^2/min) to 10 cc/min (1.348 gal/ft^2/min), the H.E.T.P. increased from 0.770 to 1.3177 inches.

Feed Volume:

It was observed that maxima of glycerol with respect to elution volume (V_m) was found to be moved forward with larger feed volumes. It was also found that as the feed volume increased, the width of the elution curves increased as tabulated in Table 4. The H.E.T.P. was found to increase linearly with feed volume on a log-log plot as shown in Figure 21. The slope of the curve was calculated to be 0.2. Evidently the dependence of feed volume on H.E.T.P. is weak. Simpson and Bauman (5) made a similar observation for ethylene glycol.

Cross-Linkage:

It was observed that the maxima of elution curves were shifted in backward direction as the cross-linkage increased. Also the width of the curves decreased. The results are tabulated in Table 5. The H.E.T.P. was found to vary slightly with cross-linkage as is apparent from the Figure 22. As the cross-linkage increased from 4 to 10 per cent the H.E.T.P. was found to increase from 2.170 to 2.740." Simpson and Bauman (5) also observed similar behaviour for ethylene glycol.

TABLE 4

EFFECT OF FEED VOLUME ON H.E.T.P.

NO.	Feed Volume cc	V_mx118 cc	Peak Glycerol concen.	W x 118 cc	W^2x118^2 cc^2	P	H.E.T.P. in.
1	5	0.64	0.177	0.1000	0.0100	32.3	.736
2	7	0.66	0.253	0.1075	0.0115	32.0	.765
3	10	0.68	0.303	0.1175	0.0138	29.5	.825
4	15	0.68	0.475	0.1200	0.0140	28.3	.864
5	20	0.72	0.615	0.1375	0.0189	25.9	.945
6	25	0.76	0.687	0.1500	0.0225	25.7	.954
7	30	0.77	0.750	0.1550	0.0240	25.0	.980
8	40	0.85	0.860	0.1900	0.0360	22.2	1.100

TABLE 5

EFFECT OF CROSS-LINKAGE ON H.E.T.P.

NO.	Cross-linkage X	V_mx118 cc	Peak Concen. of Glycerol	Wx118 cc	W^2x118^2 cc^2	P	H.E.T.P. in.
1	4	0.80	0.420	0.260	0.0675	11.2	2.170
2	8	0.72	0.495	0.225	0.0506	9.8	2.500
3	10	0.64	0.530	0.190	0.0367	9.0	2.740

Operating conditions:

(1) Temperature = 80°C
(2) Flow rate = 3.7 cc/min (0.497 gal/ft^2/min)
(3) Resin = Dowex 50W-X8, Na$^+$ form
(4) Size = 50-100 mesh
(5) Feed concentration: Sodium chloride = 2.5%W and Glycerol = 33.75%W

Slope = 0.2

V_T = 118 cm^3 (0.0040 ft^3)
h = 24.5"

H.E.T.P (inches)
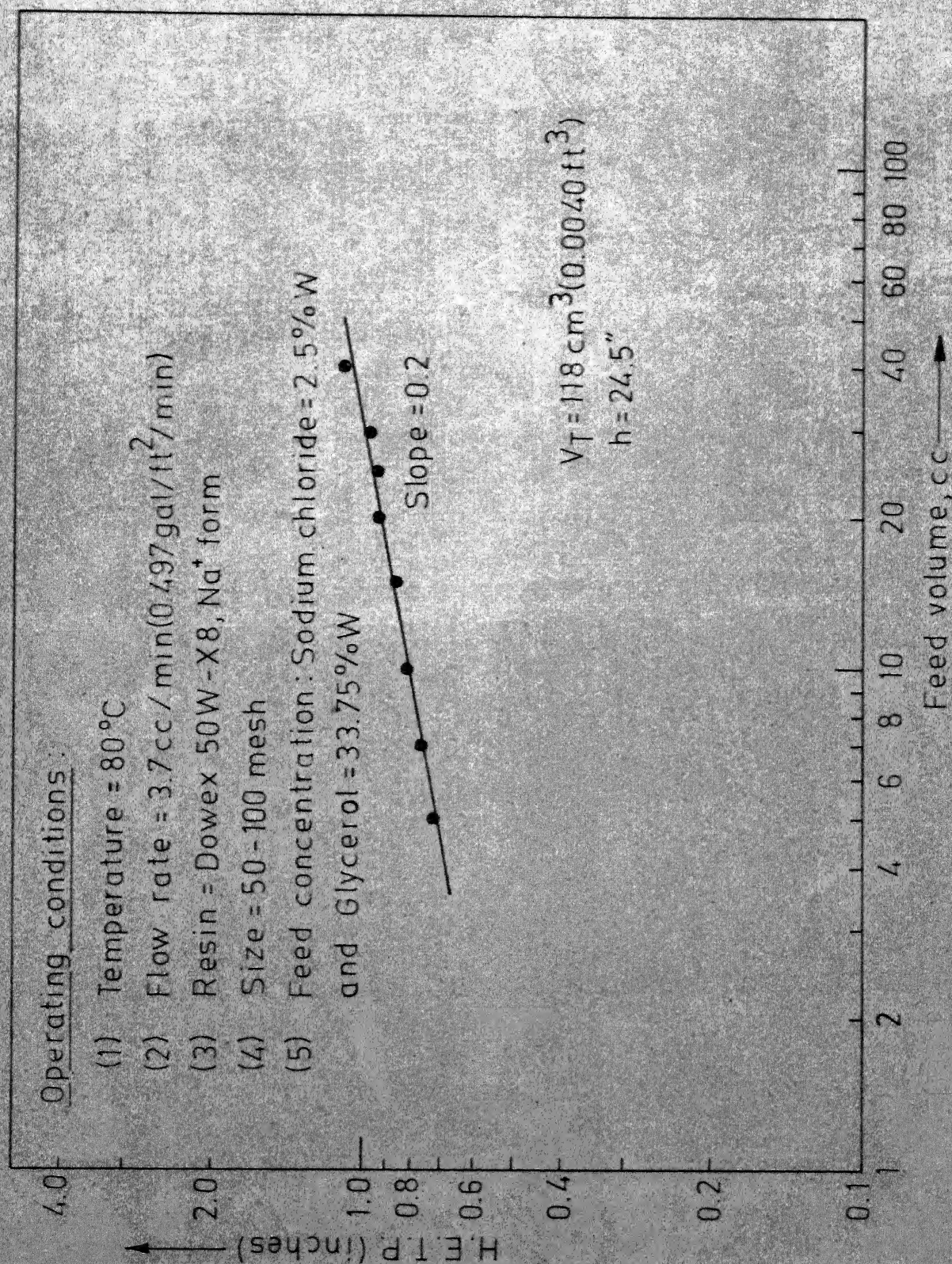
Feed volume, cc

Fig. 21 - Effect of feed volume on H.E.T.P.

Fig. 22 - Effect of cross - linkage on H.E.T.P.

Operating conditions:

(1) Temperature = 80°C

(2) Feed volume = 30 cc (25 V_T)

(3) Resin = Dowex 50W, Na^+ form

(4) Size = 20 - 50 mesh

(5) Feed con^n : Sodium chloride = 2.50%W & glycerol = 33.75%W

(6) Flow rate = 2.5 cc/min (0.337 gal/ft^2/min)

$V_T = 118 \, cm^3 (0.0040 \, ft^3)$

$h = 24.5''$

Cross - linkage of the Dowex 50W ⟶

H.E.T.P. (inches)

Particle Size:

It was observed that as the particle size increased
from 50-100 mesh to 20-50 mesh the H.E.T.P. tend to increase from
1.051 to 2.500 inches as tabulated in Table 6. Due to non-
availability of resin of other mesh sizes it was not possible
to obtain data on more than two sizes. Simpson and Bauman(5)
have reported that H.E.T.P. is directly proportional to the
particle diameter in case of ethylene glycol. It appears
reasonably to assume that similar relationship may hold good
for glycerol also.

Sodium Chloride:

As the sodium chloride concentration increased in the
feed solution the maximum concentration of glycerol was found
to increase are given in Table 7. The sodium chloride concen-
tration vs peak glycerol concentration are plotted in Figure 23
for feed volumes of 25 and 10 cc respectively. At lower sodium
chloride concentration the concentration effect on glycerol
peak concentration were insignificant but were appreciable at
higher concentration. The effect can be fully realized if a
recycle method was adopted.

TABLE 6

EFFECT OF PARTICLE SIZE ON H.E.T.P.

S. No.	Particle Size mesh	V_m x118 cc	Peak Glycerol concen.	Wx118 cc	W^2x118^2 cc^2	P	H.E.T.P. inches
1	50-100	0.72	0.770	0.1450	0.0210	23.3	1.051
2	20-50	0.72	0.495	0.2250	0.0506	9.8	2.500

TABLE 7

EFFECT OF SODIUM CHLORIDE CONCENTRATION ON
THE PEAK GLYCEROL CONCENTRATION

S. No.	Sodium Chloride concentration, per cent weight	V_m x 118 cc	Glycerol Peak concentration in the effluent
1	1.260	0.72	0.680
2	2.500	0.72	0.695
3	4.250	0.72	0.710
4	9.504	0.72	0.740
5	11.400	0.80	0.775
6	13.100	0.80	0.812
7	16.950	0.80	0.860
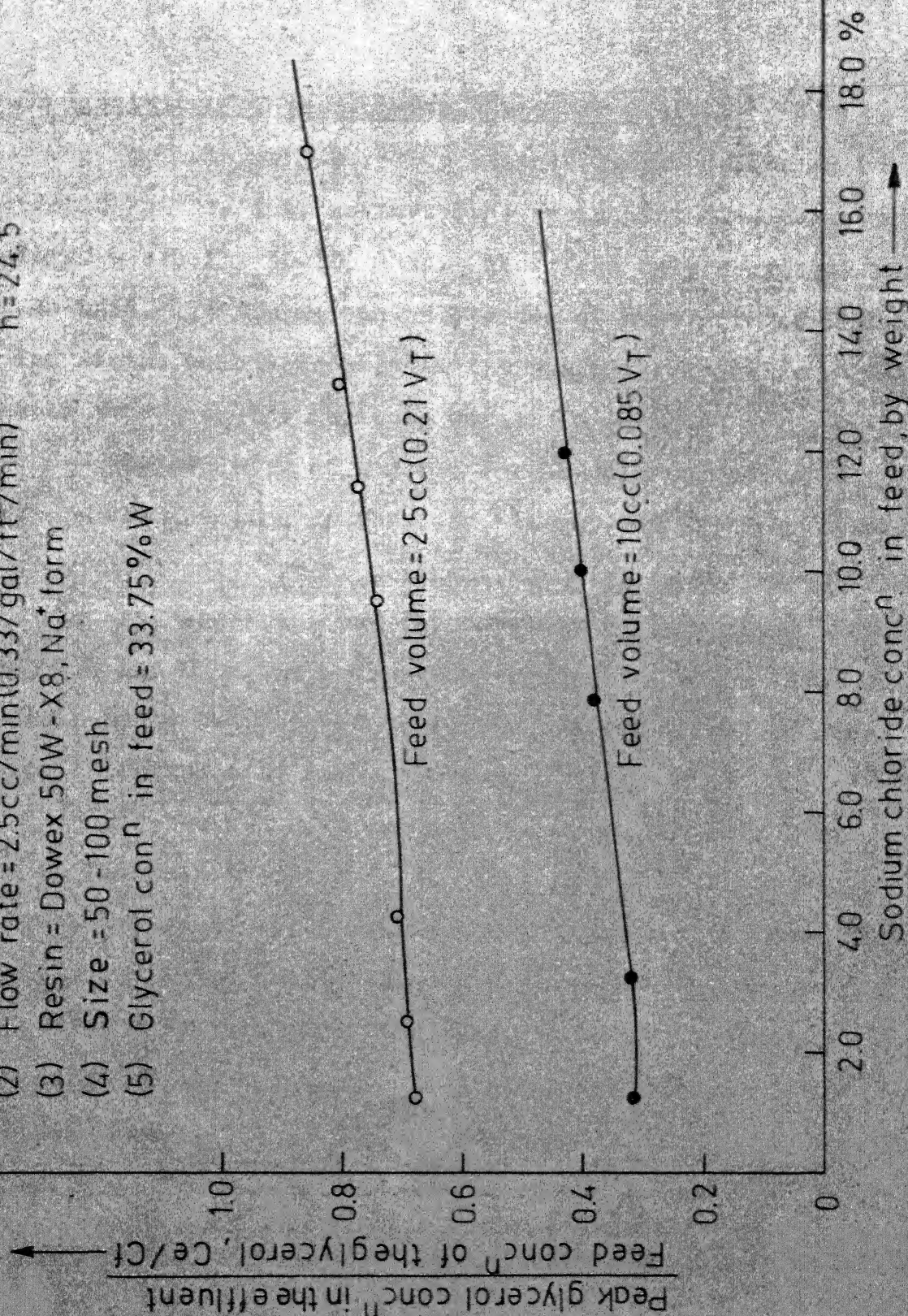
Fig. 23 - Effect of sodium chloride concentration on the glycerol concentration.
(without recycle)

VI-10 Limitation of the Ion-Exclusion Process:

The rate of adsorption of the organic molecules by the resin is very low, so that fine resin particles must be employed - usually 50-100 mesh. Therefore only very slow rates can be used. It would require the use of some sort of filter at top of the column to prevent the resin particles from being carried over with the backwash water.

The capacity of a resin for organic molecules is only a small fraction of the conventional ion exchange capacity. A sulphonated polystyrene resin of normal type has an exchange capacity of 5 meq/gram, but the capacity for adsorbing ethyl alcohol would be no more than 0.5 meq/gram. Thus a large amount of resin would be required to remove a given weight of non-ionized substances by ion-exclusion.

The resin has, of course, to be in the same ionic form as the salt to be removed. This is not difficult to achieve, and would not normally present any limitations. But, if the solution to be dealt with contains valuable ions such as gold, silver or even copper or zinc, the cost involved in conversion of the resin into the appropriate ionic form may be prohibitive.

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CHAPTER VII

CONCLUSIONS AND RECOMMENDATIONS

VII-1 Conclusion:

1. At lower temperature, both the salt wave and glycerol wave are not sharp and contamination of the product occurs. As the temperature increases the concentration and separation improved. Higher temperature thus favour good separation and high concentration. The H.E.T.P is found to decrease with increase in temperature (Figure 19).

2. At lower flow rates, the separation are better and concentrations are higher. As the flow rates increases, the contamination and dilution occurs. Lower flow rates, thus favour good separation. The H.E.T.P. is seen to be proportional to square root of the flow rate in the range studied (Figure 20).

3. At small feed volumes, the separation are good but the product is dilute. As the feed volume increases the contamination is greater, but the concentration is higher. A feed volume of $(V_e/V_T = 0.20$ to $0.40)$ was found to be optimum for a single pass operation. The H.E.T.P. is seen to increase slightly with increase in feed volume (Figure 21).

4. At lower cross-linkage, the separations are better but dilution of the product occurs. As the cross-linkage increases the separations are poor but concentrations are higher. The H.E.T.P. is found to increase slightly with increase cross-linkage (Figure 22).

5. At small particle size, the separation improves and the concentrations are found to be higher. Smaller particle size favour good separation. The H.E.T.P. is found to increase with increase in particle size.

6. At low ionic concentration the separations are good but product concentrations are lower. As ionic concentration are increased the separations are poor but the glycerol concentration improves. The glycerol peak concentration is found to be a function of sodium chloride concentration in the feed (Figure 23). However, the concentration effect are not significant unless recycle is resorted to.

7. The glycerol concentration does not have much effect on the degree of separation.

8. Using a recycle procedure it is possible to obtain good grade glycerol in concentration equal to or higher than feed concentration. The bulk of colour bodies are eluted in the salt fraction.

VII-2 Recommendations for Further Study:

The following studies are recommended in order to obtain further information.

1. To study the effect of bed height and column dimension on the separation.

2. To study the effect of sodium chloride concentration on the glycerol concentration in a recycle operation.

3. To study the recycle method for separation in greater detail from an operational view point.

4. To apply the techniques for the separation of multicomponents in a mixture. Two systems that may be of immediate interest can be mentioned (i) purification of molasses for sugar recovery; (ii) purification of invert sugar syrup made from acid hydrolysis of starchy materials.

5. To design a continuous multi bed column system.

6. To carry out bench scale and pilotplant trials for ion-exclusion technique in order to evaluate the feasibility and economics of the process.

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APPENDIX A

PROPERTIES OF THE DOWEX 50W[+]

1. Resin   Dowex 50W

2. Type   Strongly acidic cation exchanger

3. Standard cross-linkage (per cent DVB)   8

4. Special cross-linkage (per cent DVl)   1-16

5. Ionic form as supplied Na^+ or H^+ (20-50 mesh)

                                 H^+ (all other Mesh)

6. Physical form   Spheres

7. Standard mesh size (wet)   20-50

8. Special mesh size (dry)   50-100, 100-200, 200-400

9. Shipping density (lb/ft^3)

10. Moisture content (per cent)

	Na^+ form	H^+ form
	53	50
	45	53

11. Effective pH range   0-14

12. Order of selectivity for Ions $Ag > K > NH_4 > Na > H$

13. Total exchange capacity

	Na^+ form	H^+ form
Kg as $CaCO_3$/ft	41.5	37.0
Meq/g dry resin	4.8	5.0
Meq/ml wet resin	1.9	1.7

14. Spherecity (per cent)   85

15. Bed expansion   30 per cent maximum at 4 gpm/ft^2 at 25^oC

16. Pressure drop   Approximately 0.5 $lb/inch^2$

17. Stability

    Thermal   Good upto $150^{\circ}C$

    Solvent   Very good

    Oxidation   Slow solution in hot 15 per cent HNO_3

    Reduction   Very good

[+]Osborn, G.H., 'Synthetic Ion-Exchangers', pp. 36, Chapman and Hall Ltd., W.C. 2 (1961).

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APPENDIX B

DETERMINATION OF DISTRIBUTION COEFFICIENT

Bauman and Wheaton[+] have described a method for the
determination of the distribution coefficient as follows:

A known quantity of resin of known water content
is contacted with a known quantity of solution of known concen-
tration. After equilibration the new outside solution concen-
tration is determined and from this and resin volume change
the new inside concentration may be calculated. The concentration
factors are considered on a weight/volume basis assuming that
there is no intermolecular reaction between the solute and
solvent to change the total solution volume.

$$K_d = C_{s_i}/C_{s_o} = \frac{M_{s_i}}{V_{s_i}+V_{w_i}} \div \frac{M_{s_o}}{V_{s_o} + V_{w_o}}$$

$$V_{s_i} + V_{w_i} = V_i$$

$$V_{s_o} + V_{w_o} = V_o$$

$$C^o_{s_o} V^o_o - C_{s_o} V_o = C_{s_i} V_i - C^o_{s_i} V^o_i$$

where,

C^o and V^o refer to initial conditions $C^o_{s_i} V^o_i = 0$
conditions of experiment.

All terms but C_{s_i} are known

$$C_{s_i} = \frac{C_{s_o}^o V_o^o - C_{s_o} V_o}{V_i}$$

$$\frac{C_{s_i}}{C_{s_o}} = \frac{1}{V_i} \left(\frac{C_{s_o}^o V_o^o}{C_{s_o}} - V_o \right)$$

$$\frac{C_{s_i}}{C_{s_o}} = \frac{V_o}{V_i} \left(\frac{C_{s_o}^o V_o^o}{C_{s_o} V_o} \right) - 1 \right) = K_d$$

In the special case where there is no volume charge, $V_o^o = V_o$ and

$$K_d = \frac{V_o}{V_i} \left(\frac{C_{s_o}^o}{C_{s_o}} - 1 \right)$$

The concentrations may be expressed in any convenient units or physical property proportional to concentration. When refractive index (RI) is used as a measure of concentration the expression takes the form:

$$K_d = \frac{V_o}{V_i} \left[\frac{(RI_o^o - RI_w) V_o^o}{(RI_o - RI_w) V_o} \right] - 1$$

In general, for non-ionic components there is very little resin volume change except at quite high concentrations and V_i approximate to V_i^o. But with ionic materials such volume changes may be quite appreciable.

[+] Bauman, W.C., and Wheaton, R.M., Ann. N.Y. Acad. Sci., 57, 159 (1953)

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APPENDIX C

DETERMINATION OF INTERSTITIAL VOLUME OF THE RESIN[1]

A method for determining interstitial volume has been described. A 50-ml sample of resin (bulk settled volume under water) was placed in a fritted glass filter funnel after which the water was drained to the top resin level and discarded. Suction was then applied briefly to collect most of the interstitial water. Some additional water was collected by use of a centrifuge. The total volume represented V_1, V_2 was then obtained from the loss in weight after the drying the resin for 16 hours at $110^{\circ}C$. These results represent the approximate average of three determinations with individual deviations of $\pm$ 1 per cent.

V_1 and V_2 were experimentally determined by the above method and found to be 30 and 42 ml, respectively for 100 ml of Dowex 50 X-8 , 50-100 mesh.

Some data obtained with spherical resin particles are reproduced below. The particle size appears to have only a small effects upon the interstitial volume.

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[1] Wheaton, R.M., and Bauman, W.C., Ind. Eng. Chem., **45**, 228 (1953).

TABLE 8(A)

CROSS-LINKAGE VS INTERSTITIAL VOLUME[2]

Size = 200 - 400 mesh

S.No.	Resin	Interstitial volume(V_{liquid})
1.	Dowex 50-X2	0.304
2.	Dowex 50-X4	0.327
3.	Dowex 50-X8	0.379
4.	Dowex 50-X16	0.395
5.	Dowex$_1$-X2	0.351
6.	Dowex$_1$- X4	0.350
7.	Dowex$_1$- X8	0.390
8.	Dowex$_1$- X10	0.396

TABLE 8(B)

PARTICLE SIZE VS INTERSTITIAL VOLUME[3]

Resins Dowex 50-X8

S.No.	Particle Size	Interstitial Volume (V_{liquid})
1.	50-100 mesh	0.380
2.	30-40 mesh	0.370

[2] Samuelson, O., 'Ion-Exchange Separations in Analytical Chemistry', pp.128, John Wiley and Sons, New York (1963).

[3] Johnson, H.A., and Wheelock, D., I and E.O. Fundamental, 3, 201 (1964).

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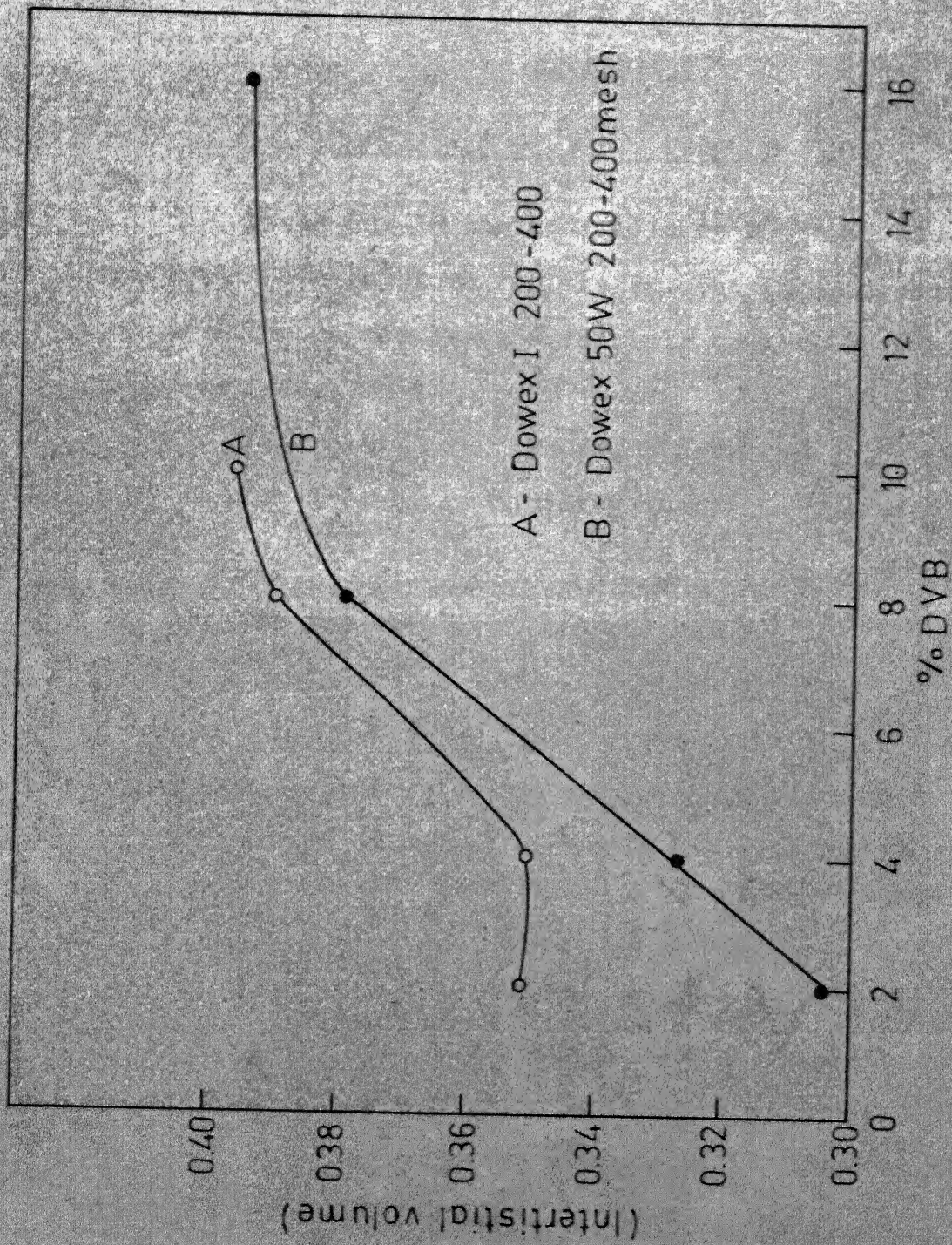
Fig. 24 - Cross-linkage vs. interstitial volume.

A - Dowex I 200-400

B - Dowex 50W 200-400mesh

APPENDIX D

CONVERSION OF IONIC FORM OF RESIN

Dowex 50W-X8 was available in H^+ form. It was converted into Na^+ form for the present investigation. The sodium chloride requirement for this conversion was calculated as given below.

Volume of Dowex 50W-X8, H^+ form = 140 cm^3

Total exchange capacity (wet volume) = 1.78 meq/ml

$\approx$ 2.00 meq/ml

For the volume of 140 cm^3 resin

= 140 x 2 = 280 meq

= 0.280 eq

Molecular weight of sodium chloride = 58.5

Weight of sodium chloride required for conversion of 140 cm^3 of Dowex 50-X8, H^+ form to Na^+ form

= 0.280 x 58.5

= 16.40 gram $\approx$ 20 gram

A 25 per cent excess was taken to ensure complete conversion.

200 ml of a 10 per cent weight sodium chloride (A.R.) was prepared. The solution was passed through the bed and eluents from the bottom were collected. The pH of the eluents were determined. The pH of the effluent at the end of operation was around 4.5-5.0, which indicate that the conversion was complete. Distilled was passed through the column to remove

excess salt and chloride ions and the effluents fractions were analysed for Cl^- by $AgNO_3$; till they gave no turbidity. The resin, completely converted into Na^+ form, was backwashed and kept ready for use.

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APPENDIX E

CALIBRATION OF THE REFRACTROMETER

For calibration of the refractrometer analytical grade of glycerol and sodium chloride were taken. The solutions were made in freshly prepared distilled water by weight percentages and the refractive indices were measured at $20^{\circ}C$. Temperature was maintained around the double prism refractrometer by circulating water from a constant temperature bath kept at this temperature.

The refractive indices of the solution were measured between the range 0-45 per cent and 0-20 per cent weight respectively for glycerol and sodium chloride. The refractive indices were also measured for the mixtures and it was observed that the refractive indices of glycerol and sodium chloride were additive. The symbol n_d^{20} is used to represent refractive index measured at $20^{\circ}C$ using light emitted by a bulb providewith the instrument.

Validity of the Additivity Rule:

n_d^{20} of distilled water $= 1.3315$

n_d^{20} of 4 per cent weight sodium chloride $= 1.3383$

n_d^{20} of 4 per cent weight glycerol $= 1.3356$

n_d^{20} of 4 per cent weight glycerol and 4 per cent sodium chloride mixture $= 1.3420$

From the additivity rule

The n_d^{20} of mixture   (4 per cent NaCl + 4 per cent glycerol)

$$= (1.3383 - 1.3315) + 1.3356$$

$$= 1.3424$$

This agrees well with the experimental value of 1.3420 . The additivity rule is thus seen to be valid for glycerol-sodium chloride-water system.

TABLE 9

COMPOSITION VS REFRACTIVE INDEX

Temperature = 20°C

Refractive index of distilled water (n_d^{20}) = 1.3315

Refractive index of glycerol (n_d^{20}) = 1.4655

S. No.	Concentration by weight per cent	Refractive index of glycerol	Refractive index of sodium chloride	Refractive index of mixture
1	2	1.3335	1.3349	1.3370
2	4	1.3356	1.3383	1.3420
3	6	1.3378	1.3415	1.3470
4	8	1.3400	1.3450	1.3535
5	10	1.3420	1.3475	1.3590
6	12	1.3445	1.3510	1.3640
7	14	1.3470	1.3550	1.3700
8	16	1.3485	1.3575	1.3745
9	18	1.3505	1.3610	1.3800
10	20	1.3525	1.3640	1.3855
11	24	1.3570	–	1.3960
12	28	1.3615	–	1.4750
13	30	1.3630	–	1.5200
14	32	1.3660	–	1.5800
15	34	1.3680	–	–
16	36	1.3700	–	–
17	40	1.3740	–	–
18	45	1.3795	–	–

Refractive index (n_d) (20°C)

Refractive index of distilled water $(n_d^{20°C}) = 1.3315$

Refractive index of glycerol $(n_d^{20°C}) = 1.4655$

(Sodium chloride + glycerol) vs. refractive index

Sodium chloride conc^n. vs. refractive index

Glycerol conc^n vs. refractive index

Conc^n. of components (wt %)
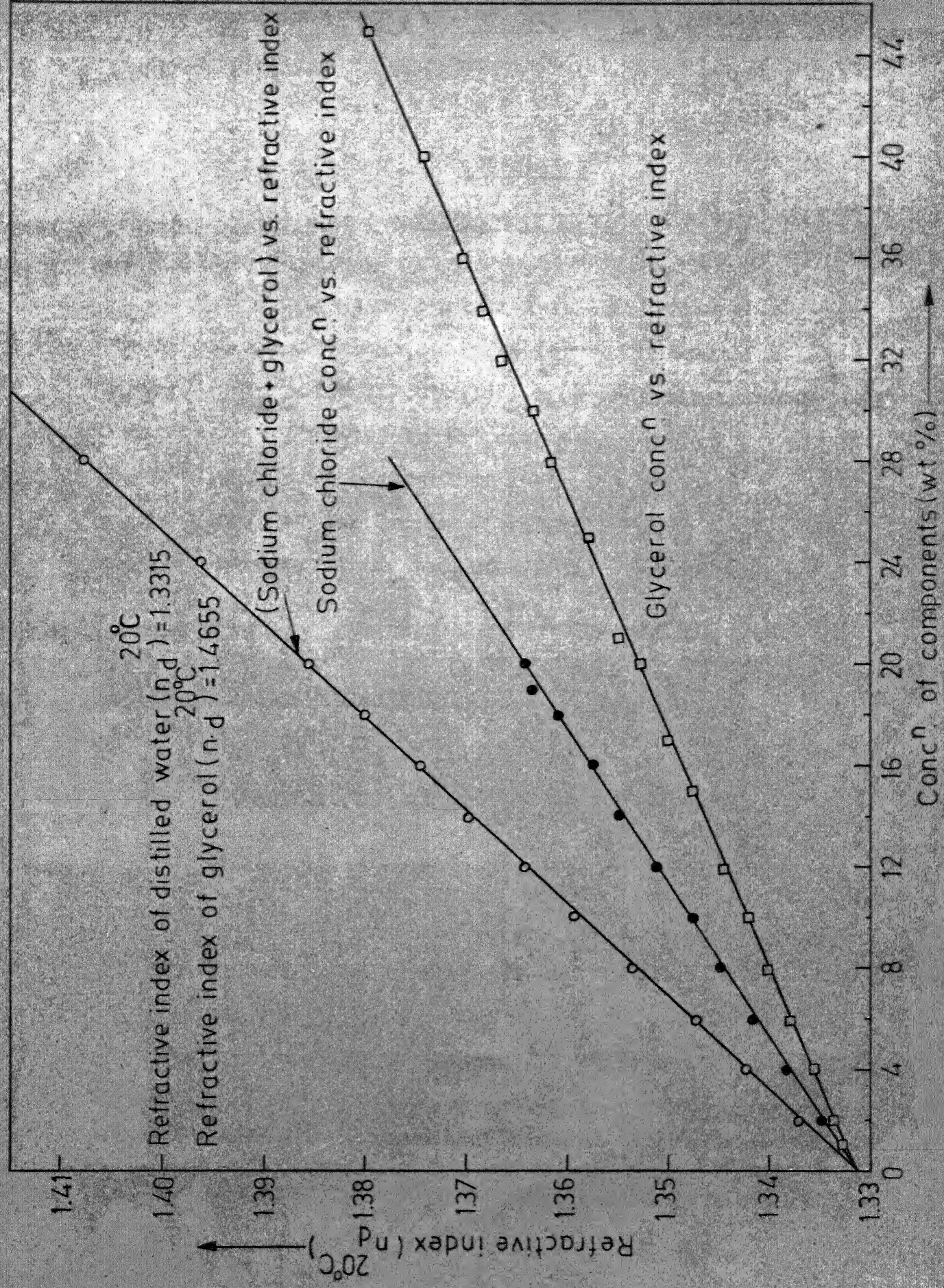
APPENDIX F

MODEL CALCULATION

Analysis:

The samples were collected in 10 ml volume and each sample was subsequently analysed for sodium chloride and glycerol. The model of calculation of analysis is shown here, Others were calculated by the same procedure.

1. n_d^{20} of the distilled water = 1.3315

2. n_d^{20} of the sample = 1.3348

3. Density of the sample = 1.013 gram/cc

4. Volume of $AgNO_3$ (0.0946 N)

   used to titrating 1 cc of sample = 3.1 cc

Sodium Chloride Concentration:

$$N_1 V_1 = N_2 V_2$$

$$1 \times N_1 = 3.1 \times 0.0946N$$

$$N_1 = 0.2935N = 0.2935 \times 58.5 = 17.169 \text{ gram/lit.}$$

$$NaCl = 17.169 \text{ gram/lit} \times \frac{1}{1.013} \text{ cc/gram} = 1.649 \text{ per cent}$$
$$\text{weight}$$

n_d^{20} of 1.649 per cent weight (from calibration curve)

$$= 1.3343$$

Glycerol Concentration:

n_d^{20} of glycerol = (1.3348 - 1.3343) + 1.3315 = 1.3320

Glycerol concentration corresponding to 1.3320 = 0.50 per cent
(from the calibration curve).   weight

Concentration Ratios:

Thus $C_{e_{NaCl}}$ = 0.2935 N ; $C_{e_{Glycerol}}$ = 0.500 per cent
                                                        weight

$$C_{f_{NaCl}} = 0.5200 \text{ N}$$

$$C_{f_{Glycerol}} = 33 \text{ per cent weight}$$

Therefore,

$$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}} = \frac{0.2935 \text{ N}}{0.5200 \text{ N}} = 0.564$$

$$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}} = \frac{0.500}{33} = 0.015$$

These ratios were calculated for each sample in this matter. The final data are given in Appendix G. The curve is plotted between V_e/V_T vs C_e/C_f. Typical plot for a run is given below.
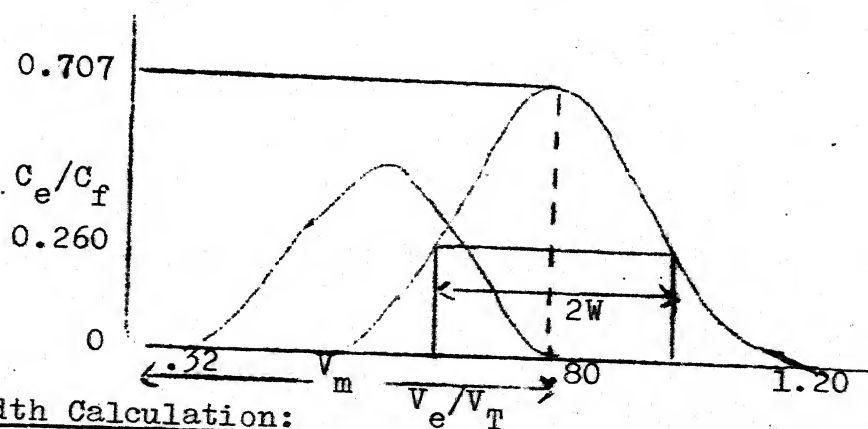
Half-Width Calculation:

$$W' = 1/e \times \text{peak concentration} = 0.368 \times 0.707 = 0.260$$

$$W = 0.1750 \times 118 \text{ cc}$$

Calculation of H.E.T.P.:

$$V_{liquid} = 38 \text{ per cent } V_T = 0.38 \times 118 \text{ cc}$$

$$V_m = 0.80 \times 118 \text{ cc}$$

$$P = \frac{2V_m(V_m - V_{liquid})}{W^2}$$

$$= \frac{2 \times 0.80 \times 118 \, (0.80 \times 118 - 0.38 \times 118)}{(0.175 \times 118)^2}$$

$$= \frac{1.60 \times 0.42}{(.175)^2} = 21.94$$

Finally,

$$H.E.T.P. = h/P = \frac{24.5}{21.94} = 1.116"$$

Values for H.E.T.P. for all the runs were obtained in this matter and hence tabulated at appropriate places.

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APPENDIX G

(EXPERIMENTAL DATA)

TABLE 10

EFFECT OF TEMPERATURE

OPERATING CONDITIONS:

1. Feed volume = 30 cc (0.25 V_T)

2. Flow rate = 2.5 cc/min (0.337 gal/ft²/min)

3. Resin = Dowex 50W-X8, Na^+ form

4. Size = 50-100 mesh

5. Feed concentration : Glycerol = 33.75 per cent W

   and sodium chloride = 2.50 per cent W (0.4128N)

$$V_T = 118 \text{ cm}^3 \ (0.0040 \text{ ft}^3)$$

$$h = 24.5"$$

EXPERIMENTAL RUN 1

Temperature = 20°C

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12 cc	–	0.0	0	0.0	0
1	10 cc	0.08	0.0	0	0.0	0
2	20 cc	0.16	0.0	0	0.0	0
3	30 cc	0.24	0.0	0	0.0	0
4	40 cc	0.32	0.0	0	0.0	0
5	50 cc	0.40	0.1585	0.385	0.0	0
6	60 cc	0.48	0.2229	0.540	1.520	0.045

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
7	70 cc	0.56	0.2640	0.640	5.675	0.167
8	80 cc	0.64	0.2279	0.552	13.900	0.410
9	90 cc	0.72	0.1320	0.320	20.250	0.600
10	100 cc	0.80	0.0820	0.200	19.400	0.575
11	110 cc	0.88	0.0495	0.120	17.700	0.525
12	120 cc	0.96	0.0206	0.050	10.500	0.310
13	130 cc	1.04	0.0	0.0	4.900	0.145
14	140 cc	1.12	0.0	0.0	1.860	0.055
15	150 cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 3

Temperature = $40^{\circ}C$

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in Normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0,0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1636	0.396	0.0	0.0
6	60cc	0.48	0.2579	0.625	1.0	0.029
7	70cc	0.56	0.2850	0.690	5.400	0.1600
8	80cc	0.64	0.2083	0.505	17.150	0.508
9	90cc	0.72	0.1140	0.276	22.250	0.660
10	100cc	0.80	0.062	0.150	20.875	0.620
11	110cc	0.88	0.0495	0.120	17.750	0.527
12	120cc	0.96	0.0048	0.011	7.600	0.225
13	130cc	1.04	0.0	0.0	2.350	0.070
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 4

Temperature = $50^{\circ}C$

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1780	0.430	0.0	0.0
6	60cc	0.48	0.2660	0.645	0.600	0.017
7	70cc	0.56	0.2900	0.700	6.750	0.200
8	80cc	0.64	0.2210	0.535	17.700	0.525
9	90cc	0.72	0.1340	0.325	21.125	0.625
10	100cc	0.80	0.0516	0.125	22.625	0.673
11	110cc	0.88	0.0206	0.050	16.250	0.482
12	120cc	0.96	0.0048	0.011	5.900	0.175
13	130cc	1.04	0.0	0.0	1.00	0.029
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 5

Temperature = 58°C

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1890	0.456	0.0	0.0
6	60cc	0.48	0.2750	0.665	0.680	0.020
7	70cc	0.56	0.2930	0.710	8.100	0.240
8	80cc	0.64	0.1840	0.445	19.100	0.565
9	90cc	0.72	0.0720	0.175	23.300	0.690
10	100cc	0.80	0.0330	0.080	21.500	0.635
11	110cc	0.88	0.0129	0.0312	12.350	0.365
12	120cc	0.96	0.0	0.0	1.9800	0.058
13	130cc	1.04	0.0	0.0	0.402	0.012
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 6

Temperature = $65^{\circ}C$

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1650	0.400	0.0	0.0
6	60cc	0.48	0.2460	0.595	1.350	0.040
7	70cc	0.56	0.2970	0.720	5.730	0.170
8	80cc	0.64	0.2311	0.560	16.900	0.500
9	90cc	0.72	0.1240	0.300	23.800	0.703
10	100cc	0.80	0.0620	0.150	22.500	0.667
11	110cc	0.88	0.0	0.0	14.500	0.430
12	120cc	0.96	0.0	0.0	4.750	0.140
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 7

Temperature = 75°C

S.No.	V_e (cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.2230	0.540	0.0	0.0
6	60cc	0.48	0.2932	0.710	1.856	0.055
7	70cc	0.56	0.3076	0.745	9.112	0.270
8	80cc	0.64	0.1899	0.460	19.912	0.590
9	90cc	0.72	0.1177	0.285	24.639	0.730
10	100cc	0.80	0.0784	0.190	22.950	0.680
11	110cc	0.88	0.0	0.0	6.412	0.190
12	120cc	0.96	0.0	0.0	0.843	0.025
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 8

Temperature = 80°C

S.No.	V_e (cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1631	0.395	0.0	0.0
6	60cc	0.48	0.2622	0.635	0.505	0.015
7	70cc	0.56	0.3097	0.750	5.062	0.150
8	80cc	0.64	0.2147	0.520	17.550	0.520
9	90cc	0.72	0.1177	0.285	25.987	0.770
10	100cc	0.80	0.0518	0.125	24.468	0.725
11	110cc	0.88	0.0	0.0	12.150	0.360
12	120cc	0.96	0.0	0.0	1.350	0.040
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

TABLE   11

EFFECT OF FLOW RATE

OPERATING CONDITIONS:

1. Temperature   = $80^{\circ}C$

2. Feed volume   = 30cc $(0.25\ V_T)$

3. Resin   = Dowex 50W – X8, Na^+ Form

4. Size   = 50-100 mesh

5. Feed concentration: Glycerol = 33.75 per cent weight

   and Sodium Chloride = 2.50 per cent weight (0.4128 N)

   V_T = 118 cm^3(0.0040 ft^3)

   h = 24.5"

EXPERIMENTAL RUN 9

Flow rate = 2.5cc/min (0.337 gal/ft^2/min)

S.No.	V_e (cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.1630	0.395	0.0	0.0
5	50cc	0.40	0.2620	0.635	0.0	0.0
6	60cc	0.48	0.3100	0.750	0.505	0.015
7	70cc	0.56	0.2140	0.520	5.060	0.150
8	80cc	0.64	0.1175	0.285	17.550	0.520

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
9	90cc	0.72	0.0516	0.125	24.470	0.725
10	100cc	0.80	0.0	0.0	26.000	0.770
11	110cc	0.88	0.0	0.0	12.150	0.360
12	120cc	0.96	0.0	0.0	13.500	0.040
13	130cc	1.04	0.0	0.0	0.00	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 10

Flow rate = 3.5cc/min(0.472 gal/ft²/min.)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in Normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (Weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.02	0.0	0.0	0.0
5	50cc	0.40	0.1280	0.310	0.0	0.0
6	60cc	0.48	0.2436	0.590	0.675	0.020
7	70cc	0.56	0.3056	0.74	4.893	0.145
8	80cc	0.64	0.2333	0.565	13.668	0.405
9	90cc	0.72	0.1177	0.285	23.456	0.695
10	100cc	0.80	0.0619	0.150	25.312	0.750
11	110cc	0.88	0.0474	0.115	14.681	0.435
12	120cc	0.96	0.0	0.0	5.062	0.150
13	130cc	1.04	0.0	0.0	1.518	0.045
14	140cc	1.12	0.0	0.0	0.506	0.015
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 11

Flow rate = 4.0 cc/min (0.540 gal/ft^2/min)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1041	0.252	0.0	0.0
6	60cc	0.48	0.2140	0.518	0.425	0.012
7	70cc	0.56	0.3024	0.732	3.92	0.116
8	80cc	0.64	0.2455	0.594	13.875	0.411
9	90cc	0.72	0.1414	0.342	22.375	0.662
10	100cc	0.80	0.1099	0.266	24.750	0.733
11	110cc	0.88	0.0392	0.094	22.625	0.670
12	120cc	0.96	0.0117	0.028	3.825	0.113
13	130cc	1.04	0.0	0.0	0.0	0.014
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 12

Flow rate = 4.45 cc/min(0.600 gal/ft^2/min)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1782	0.431	0.0	0.0
6	60cc	0.48	0.2673	0.647	0.90	0.026
7	70cc	0.56	0.3019	0.731	7.625	0.225
8	80cc	0.64	0.1980	0.479	16.750	0.496
9	90cc	0.72	0.1089	0.262	23.750	0.703
10	100cc	0.80	0.0841	0.203	24.125	0.714
11	110cc	0.88	0.0545	0.132	15.750	0.466
12	120cc	0.96	0.0	0.0	2.95	0.0874
13	130cc	1.04	0.0	0.0	1.00	0.029
14	140cc	1.12	0.0	0.0	0.09	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 13

Flow rate = 5.0cc/min(0.675 gal/ft^2/min)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1138	0.275	0.0	0.0
6	60cc	0.48	0.2377	0.575	1.012	0.030
7	70cc	0.56	0.3021	0.731	5.062	0.150
8	80cc	0.64	0.2650	0.610	12.150	0.360
9	90cc	0.72	0.1340	0.325	21.093	0.625
10	100cc	0.80	0.072	0.175	23.649	0.707
11	110cc	0.88	0.031	0.075	21.620	0.640
12	120cc	0.96	0.0	0.0	6.412	0.190
13	130cc	1.04	0.0	0.0	1.856	0.055
14	140cc	1.12	0.0	0.0	0.742	0.022
15	150cc	1.20	0.0	0.0	0.000	0.000

EXPERIMENTAL RUN 14

Flow rate = 6 cc/min(0.810 gal/ft^2/min)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1090	0.265	0.0	0.0
6	60cc	0.48	0.2350	0.570	0.202	0.006
7	70cc	0.56	0.3013	0.730	4.320	0.128
8	80cc	0.64	0.2680	0.650	11.130	0.330
9	90cc	0.72	0.1380	0.335	19.570	0.580
10	100cc	0.80	0.1200	0.290	23.000	0.680
11	110cc	0.88	0.009	0.047	21.260	0.636
12	120cc	0.96	0.0	0.0	7.700	0.230
13	130cc	1.04	0.0	0.0	2.125	0.063
14	140cc	L.12	0.0	0.0	0.470	0.014
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 15

Flow rate = 7.5 cc/min (1.012 gal/ft²/min)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1184	0.287	0.0	0.0
6	60cc	0.48	0.2447	0.570	0.850	0.025
7	70cc	0.56	0.2961	0.725	5.200	0.154
8	80cc	0.64	0.2506	0.607	11.812	0.350
9	90cc	0.72	0.1519	0.368	20.00	0.592
10	100cc	0.80	0.1085	0.220	22.500	0.666
11	110cc	0.88	0.0394	0.095	21.262	0.630
12	120cc	0.96	0.0.	0.0	8.437	0.254
13	130cc	1.04	0.0	0.0	2.362	0.070
14	140cc	1.12	0.0	0.0	0.675	0.020
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 16

Flow rate = 10cc/min (1.350 gal/ft^2/min)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.0991	0.241	0.0	0.0
6	60cc	0.48	0.2279	0.552	0.250	0.007
7	70cc	0.56	0.2973	0.720	3.575	0.106
8	80cc	0.64	0.2378	0.576	11.000	0.326
9	90cc	0.72	0.1585	0.384	19.500	0.577
10	100cc	0.80	0.1139	0.250	21.750	0.645
11	110cc	0.88	0.0819	0.130	20.875	0.618
12	120cc	0.96	0.0149	0.036	11.125	0.330
13	130cc	1.04	0.0	0.0	3.350	0.099
14	140cc	1.12	0.0	0.0	1.000	0.029
15	150cc	1.20	0.0	0.0	0.000	0.000

TABLE 12

EFFECT OF FEED VOLUME

OPERATING CONDITIONS:

1. Temperature = $80^{\circ}C$

2. Flow rate = 3.70 cc/min(0.499 gal/ft^2/min)

3. Resin = Dowex 50W-X8, Na$^+$form

4. Size = 50-100 mesh

5. Feed concentration: Glycerol = 33.75 per cent weight

and sodium chloride = 2.50 per cent weight(0.4128N)

V_T = 118 cm^3(0.0040 ft^3)

h = 24.5"

EXPERIMENTAL RUN 17

Feed Volume=5 cc(0.042V_T)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1189	0.288	0.0	0.0
6	60cc	0.48	0.0544	0.144	0.400	0.012
7	70cc	0.56	0.0	0.0	2.025	0.060

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{G_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
8	80cc	0.64	0.0	0.0	6.000	0.177
9	90cc	0.72	0.0	0.0	4.400	0.130
10	100cc	0.80	0.0	0.0	0.500	0.015
11	110cc	0.88	0.0	0.0	0.0	0.0
12	120cc	0.96	0.0	0.0	0.0	0.0
13	130cc	1.04	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 18

Feed Volume= 7 cc (0.059 V_T)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1190	0.288	0.0	0.0
6	60cc	0.48	0.1486	0.359	0.050	0.0014
7	70cc	0.56	0.0247	0.060	4.700	0.139
8	80cc	0.64	0.009	0.024	8.540	0.253
9	90cc	0.72	0.0	0.0	6.100	0.180
10	100cc	0.80	0.0	0.0	1.000	0.029
11	110cc	0.88	0.0	0.0	0.0	0.0
12	120cc	0.96	0.0	0.0	0.0	0.0
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 19

Feed volume = $10cc(0.084V_T)$

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0.	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1176	0.285	0.0	0.0
6	60cc	0.48	0.1783	0.432	0.500	0.015
7	70cc	0.56	0.0713	0.172	5.500	0.163
8	80cc	0.64	0.0198	0.047	10.250	0.304
9	90cc	0.72	0.0138	0.0336	11.875	0.352
10	100cc	0.80	0.0	0.0	3.440	0.102
11	110cc	0.88	0.0	0.0	0.500	0.015
12	120cc	0.96	0.0	0.0	0.0	0.0
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 20

Feed volume = 15 cc (0.126 V_T)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1410	0.342	0.0	0.0
6	60cc	0.48	0.2020	0.489	0.500	0.015
7	70cc	0.56	0.1190	0.288	5.880	0.174
8	80cc	0.64	0.0580	0.140	13.800	0.410
9	90cc	0.72	0.0330	0.080	16.000	0.475
10	100cc	0.80	0.0132	0.032	5.250	0.156
11	110cc	0.88	0.0	0.0	0.844	0.025
12	120cc	0.96	0.0	0.0	0.528	0.015
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 21

Feed volume = 20 cc (0.168 V_T)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1310	0.317	0.0	0.0
6	60cc	0.48	0.2370	0.575	0.500	0.015
7	70cc	0.56	0.1940	0.469	5.300	0.157
8	80cc	0.64	0.1047	0.254	16.875	0.500
9	90cc	0.72	0.0632	0.1522	20.625	0.615
10	100cc	0.80	0.0310	0.0750	18.875	0.560
11	110cc	0.88	0.0	0.0	2.450	0.072
12	120cc	0.96	0.0	0.0	0.500	0.015
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 22

Feed volume = 25 cc (0.210 V_T)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1480	0.358	0.0	0.0
6	60cc	0.48	0.2416	0.597	0.527	0.015
7	70cc	0.56	0.2820	0.682	6.850	0.203
8	80cc	0.64	0.1410	0.341	17.800	0.527
9	90cc	0.72	0.0790	0.192	23.200	0.687
10	100cc	0.80	0.0496	0.120	22.936	0.679
11	110cc	0.88	0.0222	0.053	3.611	0.107
12	120cc	0.96	0.0	0.0	1.041	0.031
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 23

Feed volume = 30 cc (0.252 V_T)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1490	0.360	0.0	0.0
6	60cc	0.48	0.2900	0.700	0.506	0.015
7	70cc	0.56	0.3096	0.750	5.737	0.170
8	80cc	0.64	0.2146	0.520	17.718	0.525
9	90cc	0.72	0.1197	0.290	24.806	0.735
10	100cc	0.80	0.0700	0.170	25.312	0.750
11	110cc	0.88	0.0395	0.095	12.150	0.360
12	120cc	0.96	0.0186	0.045	1.350	0.040
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 24

Feed volume = 40cc (0.336 V_T)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1427	0.346	0.0	0.0
6	60cc	0.48	0.2950	0.715	0.450	0.013
7	70cc	0.56	0.3282	0.795	6.625	0.196
8	80cc	0.64	0.3409	0.826	17.750	0.526
9	90cc	0.72	0.2398	0.581	24.375	0.722
10	100cc	0.80	0.1288	0.312	29.250	0.866
11	110cc	0.88	0.0725	0.175	28.500	0.844
12	120cc	0.96	0.0376	0.091	18.250	0.541
13	130cc	1.04	0.0079	0.019	1.950	0.057
14	140cc	1.12	0.0	0.0	0.500	0.015
15	150cc	1.20	0.0	0.0	0.0	0.0

TABLE 13

EFFECT OF CROSS LINKAGE

OPERATING CONDITIONS:

1. Temperature = 80°C

2. Feed volume = 30 cc (0.25 V_T)

3. Flow rate = 2.5 cc/min (0.337 gal/ft²/min)

4. Resin = Dowex 50W, Na^+ form

5. Size = 20-50 mesh

6. Feed concentration: Glycerol = 34.6 per cent weight

    and Sodium chloride = 2.60 weight per cent(0.4250N)

    V_T = 118 cm³ (0.0040 ft³)

    h = 24.5"

EXPERIMENTAL RUN 25

Cross-Linkage = X-4

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0935	0.220	5.400	0.155
5	50cc	0.40	0.1892	0.445	10.200	0.295
6	60cc	0.48	0.2365	0.555	13.500	0.390
7	70cc	0.56	0.2459	0.580	14.200	0.410

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
8	80cc	0.64	0.1870	0.440	14.600	0.420
9	90cc	0.72	0.1150	0.270	10.400	0.300
10	100cc	0.80	0.0725	0.170	7.000	0.200
11	110cc	0.88	0.0255	0.060	4.000	0.115
12	120cc	0.96	0.0.	0.0	1.910	0.055
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 26

Cross-linkage = X-8

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.1126	0.265	2.760	0.080
5	50cc	0.40	0.2252	0.530	7.100	0.205
6	60cc	0.48	0.2841	0.665	13.900	0.400
7	70cc	0.56	0.2650	0.625	16.800	0.485
8	80cc	0.64	0.1510	0.355	17.125	0.495
9	90cc	0.72	0.0640	0.150	13.250	0.385
10	100cc	0.80	0.0170	0.040	6.400	0.185
11	110cc	0.88	0.0	0.0	2.450	0.070
12	120cc	0.96	0.0	0.0	0.520	0.015
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 27

Cross-Linkage = X-10

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.1486	0.350	2.00	0.058
5	50cc	0.40	0.2477	0.585	4.180	0.120
6	60cc	0.48	0.3191	0.745	8.300	0.237
7	70cc	0.56	0.2675	0.630	15.250	0.440
8	80cc	0.64	0.1387	0.325	18.400	0.530
9	90cc	0.72	0.0510	0.120	17.500	0.505
10	100cc	0.80	0.0	0.0	12.375	0.355
11	110cc	0.88	0.0	0.0	2.950	0.085
12	120cc	0.96	0.0	0.0	1.000	0.029
13	130cc	1.04	0.0	0.0	0.000	0.000
14	140cc	1.12	0.0	0.0	0.000	0.000
15	150cc	1.20	0.0	0.0	0.000	0.000

TABLE 14

EFFECT OF PARTICLE SIZE

OPERATING CONDITIONS:

1. Temperature = $80^{o}C$

2. Feed volume = 30 cc(0.25 V_T)

3. Flow rate = 2.5 cc/min (0.337 gal/ft^2/min)

4. Resin =n Dowex 50W-X8, Na$^+$ form

5. Feed concentration: Glycerol = 34.6 per cent weight

and Sodium chloride = 2.60 per cent weight(0.4250N)

V_T = 118 cm^3 (0.0040 ft^3)

h = 24.5"

EXPERIMENTAL RUN 28

Particle Size = 20 - 50 mesh

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.1126	0.265	2.760	0.080
5	50cc	0.40	0.2250	0.530	7.100	0.205
6	60cc	0.48	0.2841	0.665	13.900	0.400
7	70cc	0.56	0.2650	0.625	16.800	0.485

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
8	80cc	0.64	0.1510	0.355	17.125	0.495
9	90cc	0.72	0.0640	0.150	13.350	0.385
10	100cc	0.80	0.0170	0.040	6.400	0.185
11	110cc	0.88	0.0	0.0	2.450	0.070
12	120cc	0.96	0.0	0.0	0.520	0.015
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 29

Particle Size=50-100mesh

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1678	0.395	0.0	0.0
6	60cc	0.48	0.2698	0.635	0.520	0.0150
7	70cc	0.56	0.3187	0.750	5.200	0.150
8	80cc	0.64	0.2210	0.520	17.992	0.520
9	90cc	0.72	0.1211	0.285	26.642	0.770
10	100cc	0.80	0.0531	0.125	25.085	0.725
11	110cc	0.88	0.0	0.0	12.456	0.360
12	120cc	0.96	0.0	0.0	1.384	0.040
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

TABLE 15(A)

EFFECT OF SODIUM CHLORIDE CONCENTRATION

OPERATING CONDITIONS:

1. Temperature = $80^{\circ}C$

2. Flow rate = 2.5 cc/min (0.337 gal/ft^2/min)

3. Feed volume = 25 cc (0.210 V_T)

4. Resin = Dowex 50W-X8, Na$^+$ form

5. Size = 50-100 mesh

6. Feed concentration: Glycerol = 33.75 per cent weight

$$V_T = 118 \text{ cm}^3 (0.0040 \text{ ft}^3)$$
$$h = 24.5"$$

EXPERIMENTAL RUN 30

Sodium chloride concentration = 1.260 per cent weight
(0.2357 N)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1188	0.504	0.0	0.0
6	60cc	0.48	0.1584	0.672	1.933	0.057
7	70cc	0.56	0.1089	0.462	10.732	0.318

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
8	80cc	0.64	0.0643	0.272	20.418	0.605
9	90cc	0.72	0.0400	0.170	22.950	0.680
10	100cc	0.80	0.0237	0.100	15.350	0.455
11	110cc	0.88	0.009	0.041	2 .413	0.071
12	120cc	0.96	0.0	0.0	1.012	0.030
13	130cc	1.04	0.0	0.0	0.500	0.0150
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 31

Sodium chloride concentration=2.50 weight per cent
(0.4128N)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1461	0.354	0.0	0.0
6	60cc	0.48	0.2476	0.600	1.856	0.055
7	70cc	0.56	0.2786	0.675	6.885	0.204
8	80cc	0.64	0.1450	0.350	18.225	0.540
9	90cc	0.72	0.0916	0.222	23.456	0.695
10	100cc	0.80	0.0531	0.130	22.275	0.660
11	110cc	0.88	0.0206	0.050	2.700	0.080
12	120cc	0.96	0.0	0.0	0.338	0.0100
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 32

Sodium chloride concentration = 4.25 per cent weight(0.8110N)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.2871	0.354	0.0	0.0
6	60cc	0.48	0.4999	0.615	1.687	0.050
7	70cc	0.56	0.5544	0.681	6.885	0.204
8	80cc	0.64	0.3168	0.391	18.900	0.560
9	90cc	0.72	0.1980	0.244	23.960	0.710
10	100cc	0.80	0.1216	0.150	21.532	0.638
11	110cc	0.88	0.0486	0.060	3.375	0.100
12	120cc	0.96	0.0	0.0	1.350	0.040
13	130cc	1.04	0.0	0.0	0.500	0.015
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 33

Sodium chloride concentration = 9.50 per cent weight(1.877N)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.5630	0.300	0.0	0.0
6	60cc	0.48	0.9529	0.505	1.518	0.045
7	70cc	0.56	1.2571	0.670	6.075	0.180
8	80cc	0.64	0.8491	0.450	16.875	0.500
9	90cc	0.72	0.6604	0.350	25.000	0.740
10	100cc	0.80	0.3113	0.165	23.640	0.700
11	110cc	0.88	0.0938	0.050	6.750	0.200
12	120cc	0.96	0.0	0.0	2.020	0.060
13	130cc	1.04	0.0	0.0	0.506	0.015
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 34

Sodium chloride concentration = 11.400 per cent weight(2.279N)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.4850	0.212	0.0	0.0
6	60cc	0.48	0.9800	0.430	1.316	0.039
7	70cc	0.56	1.3800	0.605	4.725	0.140
8	80cc	0.64	1.4500	0.635	14.512	0.430
9	90cc	0.72	0.9000	0.395	23.962	0.710
10	100cc	0.80	0.4150	0.180	26.156	0.775
11	110cc	0.88	0.1823	0.080	8.775	0.260
12	120cc	0.96	0.0455	0.020	2.531	0.070
13	130cc	1.04	0.0	0.0	0.526	0.015
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 36

Sodium chloride concentration = 16.95 per cent weight(3.460N)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.3633	0.105	0.0	0.0
6	60cc	0.48	0.7820	0.225	1.020	0.030
7	70cc	0.56	1.6690	0.482	2.700	0.080
8	80cc	0.64	2.160	0.625	11.500	0.340
9	90cc	0.72	2.200	0.635	24.500	0.725
10	100cc	0.80	0.9653	0.275	29.000	0.860
11	110cc	0.88	0.3820	0.110	19.100	0.565
12	120cc	0.96	0.1033	0.030	4.050	0.120
13	130cc	1.04	0.0	0.0	0.844	0.025
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

TABLE 15(B)

EFFECT OF SODIUM CHLORIDE CONCENTRATION

OPERATING CONDITIONS:

1. Temperature = 80°C

2. Flow rate = 2.5 cc/min (0.337 gal/ft^2/min)

3. Feed volume = 10 cc (0.084 V_T)

4. Resin = Dowex 50W-X8, Na$^+$ form

5. Size = 50-100 mesh

6. Feed concentration: Glycerol = 33.75 per cent weight

$$V_T = 118 \text{ cm}^3/ \ (0.0040 \text{ ft}^3)$$
$$h = 24.5"$$

EXPERIMENTAL RUN 37

Sodium chloride concentration=1.262 per cent weight
(0.2357N)

S.No.	V_e (Cumula-tive)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in norma-lity)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0130	0.055	0.0	0.0
5	50cc	0.40	0.0777	0.330	0.0	0.0
6	60cc	0.48	0.0814	0.345	0.337	0.010
7	70cc	0.56	0.0235	0.100	4.218	0.125

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
8	80cc	0.64	0.0094	0.040	10.631	0.315
9	90cc	0.72	0.0047	0.020	10.293	0.305
10	100cc	0.80	0.0	0.0	1.687	0.050
11	110cc	0.88	0.0	0.0	0.506	0.015
12	120cc	0.96	0.0	0.0	0.0	0.0
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 38

Sodium chloride concentration = 3.30 per cent weight
(0.6284N)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0314	0.050	0.0	0.0
5	50cc	0.40	0.1853	0.295	0.0	0.0
6	60cc	0.48	0.2168	0.340	0.506	0.015
7	70cc	0.56	0.0785	0.1250	4.220	0.125
8	80cc	0.64	0.031	0.050	10.968	0.325
9	90cc	0.72	0.0157	0.025	10.800	0.320
10	100cc	0.80	0.0	0.0	2.362	0.070
11	110cc	0.88	0.0	0.0	1.350	0.040
12	120cc	0.96	0.0	0.0	0.675	0.020
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 39

Sodium chloride concentration = 7.50 per cent weight
(1.414 N)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0565	0.040	0.0	0.0
5	50cc	0.40	0.2828	0.200	0.0	0.0
6	60cc	0.48	0.4949	0.350	0.338	0.010
7	70cc	0.56	0.2898	0.205	3.375	0.100
8	80cc	0.64	0.0919	0.065	12.487	0.370
9	90cc	0.72	0.0466	0.033	10.968	0.325
10	100cc	0.80	0.0	0.0	2.025	0.060
11	110cc	0.88	0.0	0.0	0.675	0.020
12	120cc	0.96	0.0	0.0	0.337	0.010
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 40

Sodium chloride concentration = 10.0 per cent weight(1.8873N)

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0751	0.040	0.0	0.0
5	50cc	0.40	0.3302	0.175	0.0	0.0
6	60cc	0.48	0.6794	0.360	0.337	0.010
7	70cc	0.56	0.5660	0.300	2.362	0.670
8	80cc	0.64	0.1890	0.100	13.500	0.400
9	90cc	0.72	0.0566	0.030	12.990	0.380
10	100cc	0.80	0.0	0.0	2.868	0.085
11	110cc	0.88	0.0	0.0	0.843	0.025
12	120cc	0.96	0.0	0.0	0337	0.010
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 41

Sodium chloride concentration =12 per cent weight(2.264N)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0565	0.025	0.0	0.0
5	50cc	0.40	0.2604	0115	0.0	0.0
6	60cc	0.48	0.6452	0.285	0.337	0.010
7	70cc	0.56	0.8376	0.370	0.678	0.020
8	80cc	0.64	0.2943	0.130	11.137	0.330
9	90cc	0.72	0.1018	0.045	14.512	0.430
10	100cc	0.80	0.0026	0.010	3.206	0.095
11	110cc	0.88	0.0	0.0	1.012	0.030
12	120cc	0.96	0.0	0.0	0.050	0.015
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

TABLE 16

EFFECT OF GLYCEROL CONCENTRATION

OPERATING CONDITIONS:

1. Temperature = $80^\circ C$

2. Flow rate = 2.5 cc/min (0.337 gal/ft^2/min)

3. Feed volume = 30 cc(0.25 V_T)

4. Resin = Dowex 50W - X8, Na$^+$ form

5. Size = 50-100 mesh

6. Feed concentration: Sodium chloride = 2.50 per cent
   weight(0.4128N)

$$V_T = 118 \text{ cm}^3 \text{ (0.0040 ft}^3)$$
$$h = 24.5"$$

EXPERIMENTAL RUN 42

Glycerol concentration=6.75 percent
weight

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12oc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0290	0.070	0.0	0.0
5	50cc	0.40	0.1570	0.383	0.0	0.0
6	60cc	0.48	0.2800	0.680	0.500	0.074
7	70cc	0.56	0.3350	0.810	1.210	0.180

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	C_e (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
8	80cc	0.64	0.3050	0.740	2.500	0.370
9	90cc	0.72	0.0262	0.0635	6.420	0.940
10	100cc	0.80	0.0180	0.0435	5.400	0.800
11	110cc	0.88	0.0	0.0	1.500	0.222
12	120cc	0.96	0.0	0.0	0.500	0.074
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 43

Glycerol=10.625
per cent weight

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0610	0.148	0.0	0.0
5	50cc	0.40	0.2030	0.490	0.0	0.0
6	60cc	0.48	0.2960	0.720	0.080	0.0750
7	70cc	0.56	0.3140	0.760	4.150	0.3900
8	80cc	0.64	0.1840	0.446	8.550	0.8000
9	90cc	0.72	0.0825	0.200	9.800	0.920
10	100cc	0.80	0.0412	0.100	8.550	0.800
11	110cc	0.88	0.0124	0.030	2.130	0.200
12	120cc	0.96	0.0	0.0	0.500	0.047
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 44

Glycerol = 16.250 weight per cent

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0527	0.1275	0.0	0.0
5	50cc	0.40	0.1850	0.450	0.0	0.0
6	60cc	0.48	0.2950	0.715	0.602	0.037
7	70cc	0.56	0.3150	0.765	5.200	0.320
8	80cc	0.64	0.1840	0.448	11.500	0.710
9	90cc	0.72	0.1050	0.255	14.200	0.870
10	100cc	0.80	0.0518	0.125	12.300	0.753
11	110cc	0.88	0.0175	0.0425	4.000	0.245
12	120cc	0.96	0.0	0.0	0.490	0.030
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 45

Glycerol Concentration=21.875 per cent
weight

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0695	0.169	0.0	0.0
5	50cc	0.40	0.2100	0.510	0.243	0.0115
6	60cc	0.48	0.300	0.724	1.8400	0.084
7	70cc	0.56	0.3100	0.750	8.700	0.400
8	80cc	0.64	0.1700	0.425	16.900	0.770
9	90cc	0.72	0.1050	0.255	18.400	0.840
10	100cc	0.80	0.0523	0.127	17.900	0.815
11	110cc	0.88	0.0206	0.050	4.400	0.202
12	120cc	0.96	0.0	0.0	0.530	0.250
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 46

Glycerol Concentration=43.2 per cent weight

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0350	0.085	0.0	0.0
5	50cc	0.40	0.1940	0.470	0.0	0.0
6	60cc	0.48	0.2900	0.700	0.0250	0.0058
7	70cc	0.56	0.3170	0.770	0.242	0.0554
8	80cc	0.64	0.2050	0.500	14.500	0.335
9	90cc	0.72	0.1130	0.275	29.000	0.670
10	100cc	0.80	0.0600	0.145	34.600	0.800
11	110cc	0.88	0.0178	0.0425	30.400	0.700
12	120cc	0.96	0.0	0.0	18.800	0.432
13	130cc	1.04	0.0	0.0	6.500	0.150
14	140cc	1.12	0.0	0.0	0.000	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 47

Glycerol concentration=51.40 per cent
weight

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0700	0.169	0.0	0.0
5	50cc	0.40	0.2100	0.510	0.247	0.0048
6	60cc	0.48	0.3100	0.750	3.280	0.0635
7	70cc	0.56	0.3240	0.784	15.900	0.310
8	80cc	0.64	0.1750	0.425	35.300	0.687
9	90cc	0.72	0.1050	0.255	38.000	0.740
10	100cc	0.80	0.0618	0.1490	24.700	0.480
11	110cc	0.88	0.0288	0.070	8.220	0.160
12	120cc	0.96	0.0	0.0	1.500	0.0292
13	130cc	1.04	0.0	0.0	0.0	0.0
14	140cc	1.12	0.0	0.0	0.0	0.0
15	150cc	1.20	0.0	0.0	0.0	0.0

TABLE 17

EFFECT OF RECYCLE

OPERATING CONDITIONS:

1. Temperature = 80°C

2. Flow rate = 2.5 cc/min (0.337 gal/ft²/min)

3. Resin = Dowex 50W-X8, Na⁺ form

4. Size = 50-100 mesh

5. Feed concentration: Glycerol =33.75 per cent weight

   and sodium chloride = 2.86 per cent weight
   (0.5200N)

$$V_T = 118 \text{ cm}^3 (0.0040 \text{ ft}^3)$$
$$h = 24.5"$$

EXPERIMENTAL RUN 48

(i) Recycle: Without

(ii) Feed = 40 cc (0.336 V_T)

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.2365	0.455	0.0	0.0
6	60cc	0.48	0.3760	0.725	1.325	0.041
7	70cc	0.56	0.4360	0.840	8.750	0.265

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
8	80cc	0.64	0.4540	0.870	17.750	0.538
9	90cc	0.72	0.2338	0.460	25.625	0.780
10	100cc	0.80	0.1140	0.220	28.000	0.850
11	110cc	0.88	0.0468	0.090	24.500	0.742
12	120cc	0.96	0.0	0.0	7.125	0.215
13	130cc	1.04	0.0	0.0	1.675	0.050
14	140cc	1.12	0.0	0.0	0.500	0.014
15	150cc	1.20	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 49

(i) Recycle: First

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.1135	0.2170	0.0	0.0
6	60cc	0.48	0.1990	0.3830	0.0	0.0
7	70cc	0.56	0.2838	0.5450	0.750	0.0227
8	80cc	0.64	0.3480	0.6700	1.625	0.0495
9	90cc	0.72	0.4080	0.7850	4.075	0.1238
10	100cc	0.80	0.4450	0.8570	8.375	0.2540
11	110cc	0.88	0.4830	0.9300	16.250	0.495
12	120cc	0.96	0.4830	0.9300	24.750	0.750
13	130cc	1.04	0.4430	0.852	28.375	0.860
14	140cc	1.12	0.3400	0.6540	29.750	0.205
15	150cc	1.20	0.2360	0.455	30.625	0.935
16	160cc	1.28	0.0570	0.100	28.625	0.920
17	170cc	1.36	0.0	0.0	27.875	0.841

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
18	180cc	1.44	0.0	0.0	22.875	0.690
19	190cc	1.52	0.0	0.0	11.000	0.3350
20	200cc	1.60	0.0	0.0	4.225	0.1280
21	210cc	1.68	0.0	0.0	1.000	0.0303
22	220cc	1.76	0.0	0.0	0.0	0.0
23	230cc	1.84	0.0	0.0	0.0	0.0
24	240cc	1.92	0.0	0.0	0.0	0.0
25	250cc	2.00	0.0	0.0	0.0	0.0

EXPERIMENTAL RUN 50

(i) Recycle: Second

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.0750	0.1460	0.0	0.0
6	60cc	0.48	0.1513	0.2900	0.0	0.0
7	70cc	0.56	0.2270	0.4370	0.55	0.0161
8	80cc	0.64	0.2932	0.5650	0.60	0.0182
9	90cc	0.72	0.3500	0.6720	1.10	0.0333
10	100cc	0.80	0.3878	0.7380	1.675	0.0505
11	110cc	0.88	0.4162	0.800	3.375	0.102
12	120cc	0.96	0.4351	0.840	6.025	0.183
13	130cc	1.04	0.4830	0.930	10.000	0.303
14	140cc	1.12	0.4830	0.930	14.750	0.445
15	150cc	1.20	0.4830	0.9300	20.500	0.621

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
16	160cc	1.28	0.4830	0.9300	26.250	0.805
17	170cc	1.36	0.4446	0.8555	29.875	0.905
18	180cc	1.44	0.4250	0.820	30.625	0.931
19	190cc	1.52	0.3878	0.748	31.600	0.960
20	200cc	1.60	0.1608	0.1608	32.000	0.970
21	210cc	1.68	0.0	0.0	32.400	0.980
22	220cc	1.76	0.0	0.0	32.000	0.970
23	230cc	1.84	0.0	0.0	31.600	0.960
24	240cc	1.92	0.0	0.0	28.000	0.850
25	250cc	2.00	0.0	0.0	20.000	0.608
26	260cc	2.08	0.0	0.0	9.125	0.277
27	270cc	2.16	0.0	0.0	3.450	0.105
28	280cc	2.24	0.0	0.0	1.000	0.0303
29	290cc	2.32	0.0	0.0	0.500	0.0157
30	300cc	2.40	0.0	0.0	0.000	0.0000
31	310cc	2.48	0.0	0.0	0.000	0.0000

EXPERIMENTAL RUN 51

(i) Recycle: Third

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0	0.0	0.0	0.0
5	50cc	0.40	0.0757	0.1450	0.0	0.0
6	60cc	0.48	0.1325	0.254	0.225	0.0068
7	70cc	0.56	0.1799	0.345	0.275	0.0083
8	80cc	0.64	0.2367	0.455	0.500	0.0151
9	90cc	0.72	0.2935	0.5644	0.600	0.0180
10	100cc	0.80	0.3314	0.6373	0.800	0.0240
11	110cc	0.88	0.3788	0.7284	0.900	0.027
12	120cc	0.96	0.4072	0.7830	2.025	0.061
13	130cc	1.04	0.4450	0.855	3.100	0.093
14	140cc	1.12	0.4735	0.910	6.250	0.1893
15	150cc	1.20	0.4829	0.928	10.625	0.3219
16	160cc	1.28	0.4924	0.946	14.875	0.4507

S.No.	V_e (Cumulative)	$\frac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\frac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\frac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
17	170cc	1.36	0.5019	0.9651	19.250	0.583
18	180cc	1.44	0.5208	1.000	24.875	0.7537
19	190cc	1.52	0.5208	1.000	27.750	0.8409
20	200cc	1.60	0.5208	1.000	30.800	0.9330
21	210cc	1.68	0.4840	0.930	32.000	0.9700
22	220cc	1.76	0.4450	0.855	32.200	0.9800
23	230cc	1.84	0.3693	0.710	32.800	0.9970
24	240cc	1.92	0.1136	0.2184	33.200	1.0140
25	250cc	2.00	0.0	0.0	33.200	1.0140
26	260cc	2.08	0.0	0.0	32.800	0.9950
27	270cc	2.16	0.0	0.0	32.700	0.9940
28	280cc	2.24	0.0	0.0	32.600	0.9930
29	290cc	2.32	0.0	0.0	31.600	0.2600
30	300cc	2.40	0.0	0.0	23.75	0.7190
31	310cc	2.48	0.0	0.0	13.75	0.4176
32	320cc	2.56	0.0	0.0	5.75	0.1742
33	330cc	2.64	0.0	0.0	2.35	0.0712
34	340cc	2.72	0.0	0.0	0.50	0.0148
35	350cc	2.80	0.0	0.0	0.00	0.0000
36	360cc	2.88	0.0	0.0	0.00	0.0000
37	370cc	2.96	0.0	0.0	0.00	0.0000
38	380cc	3.04	0.0	0.0	0.00	0.0000

EXPERIMENTAL RUN 52

(i) Recycle: Fourth

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
0	12cc	0.0	0.0	0.0	0.0	0.0
1	10cc	0.08	0.0	0.0	0.0	0.0
2	20cc	0.16	0.0	0.0	0.0	0.0
3	30cc	0.24	0.0	0.0	0.0	0.0
4	40cc	0.32	0.0756	0.1450	0.25	0.0075
5	50cc	0.40	0.2270	0.435	0.70	0.0212
6	60cc	0.48	0.3216	0.620	1.425	0.0432
7	70cc	0.56	0.3973	0.765	2.050	0.0621
8	80cc	0.64	0.4351	0.835	3.725	0.1130
9	90cc	0.72	0.4730	0.910	5.875	0.1780
10	100cc	0.80	0.4824	0.930	8.125	0.2460
11	110cc	0.88	0.4824	0.930	12.750	0.3870
12	120cc	0.96	0.5108	0.980	16.875	0.5110
13	130cc	1.04	0.5108	0.980	20.375	0.6170
14	140cc	1.12	0.5108	0.980	23.375	0.7070
15	150cc	1.20	0.5108	0.980	28.375	0.8600
16	160cc	1.28	0.5108	0.980	30.600	0.928

S.No.	V_e (Cumulative)	$\dfrac{V_e}{V_T}$	$C_{e_{NaCl}}$ (in normality)	$\dfrac{C_{e_{NaCl}}}{C_{f_{NaCl}}}$	$C_{e_{Glycerol}}$ (weight per cent)	$\dfrac{C_{e_{Glycerol}}}{C_{f_{Glycerol}}}$
17	170cc	1.36	0.4824	0.930	31.600	0.956
18	180cc	1.44	0.4730	0.910	32.600	0.988
19	190cc	1.52	0.4446	0.856	32.800	0.995
20	200cc	1.60	0.3784	0.725	32.800	0.995
21	210cc	1.68	0.1513	0.291	32.800	0.995
22	220cc	1.76	0.0378	0.073	33.000	1.000
23	230cc	1.84	0.0	0.0	33.700	1.008
24	240cc	1.92	0.0	0.0	32.800	0.995
25	250cc	2.00	0.0	0.0	32.750	0.992
26	260cc	2.08	0.0	0.0	32.750	0.992
27	270cc	2.16	0.0	0.0	32.750	0.992
28	280cc	2.24	0.0	0.0	32.200	0.975
29	290cc	2.32	0.0	0.0	27.600	0.838
30	300cc	2.40	0.0	0.0	17.500	0.530
31	310cc	2.48	0.0	0.0	7.125	0.216
32	320cc	2.56	0.0	0.0	2.000	0.0605
33	330cc	2.64	0.0	0.0	0.000	0.0000
34	340cc	2.72	0.0	0.0	0.000	0.0000
35	350cc	2.80	0.0	0.0	0.000	0.0000
36	360cc	2.88	0.0	0.0	0.000	0.0000
37	370cc	2.96	0.0	0.0	0.000	0.0000
38	380cc	3.04	0.0	0.0	0.000	0.0000